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Food Technology and Defense

The I. F. T. should never be forgetful of its responsibilities to the nation. Every war or possibility of future war brings new problems to those responsible for food supply for either offense or defense. All who are old enough to recall the situations which existed previous to each of the two World Wars can remember the confusion and the general interference, by presumably patriotic but often irresponsible purveyors of unproved products, with the hurried and not always clear-cut plans and methods of procedure which perhaps necessarily characterized the national unpreparedness that existed in each of these periods. In each, there was a question of obtaining sufficient quantities of food materials, and of assurance of technological and nutritional fitness for the special needs and ability to meet the requirements of the established military rations. A situation of this sort might happen again.

While this may be a condition that cannot be completely or possibly largely forestalled, the delay might be considerably minimized by planned cooperative assistance by competent scientific groups. None is as competent as the Institute of Food Technologists in clearly recognizing the great importance of food as a munition of war, both for offense and defense, and our organization might well organize its thinking and its objectives in such a way as to be of greatest and most immediate service in case the emergency should suddenly arise. Actual combat to protect one's home or land from attack is merely one simple form of defense. A larger and more important one is concerned with the provision of proper supplies for all divisions of the armed forces—the army, navy, air force, and the training camps, and for the associated ranks of the Red Cross, hospitals, and the thousands of non-combatants required in maintenance and in centers of supply on the home front. This might also include the feeding of workers in industrial plants commandeered for war work. Moreover, the same types of foods could meet the needs of much of our civilian population even in "the piping times of peace."

World War II taught us the necessity for prompt action. It also taught us that if we would avoid great waste, foods for defense must be nutritionally satisfactory both in quality and in acceptability. We learned also that they must be prepared not only against ordinary spoilage and deterioration but they must be able to withstand Arctic cold and Torrid heat without loss of value, and must be so packaged as to protect against moisture infiltration, insect and fungus attack, poison gas, rust or chemical deterioration, or the ravages of time. Much progress in most of these matters was attained during the latest war, but it was done the hard way and the costly way. Great service could be done for foods for general use by planned research along lines which were then begun.

There are probably innumerable "confidential" reports of value buried in official files. It seems that it might be wise to review and evaluate these, and to collect and collate the results of the experience of the past ten years, and see if some way to make new progress may not be derived from their study.

It seems not unrealistic to think that such a review, carefully studied and digested, might suggest some leads toward a more integrated relation between foods for defense and foods fully acceptable for commercial distribution and broad civilian consumption. After all, the consumers all have the same nutritive requirements, and approximately similar tastes. Moreover, the buying public is already familiar with many types of concentrated or condensed foods.

Here is also a posibility that might interest the broad-minded and far-seeing food manufacturer who appreciates what the trained food technologist might do for him by research, and by scientific methods of preparation, production, and control.

A survey of the troop-feeding programs in the United States Army from 1775 to 1940 showed the vast change in the ration in the long period up to and including the Civil War and the more humanized and nutritionally adequate ration of the World War II period which made the U. S. Army the best-fed group of our citizens. Principles and practices to be worked out by food technologists might bring defense foods and many special lines equally adaptable to adequate civilian consumption into the same general class. Then, at least, the soldier would not have the mistaken idea that he was receiving inferior food.

Individual members of I. F. T. are already cooperating officially with the Quartermaster Board, the Food and Container Institute, and other Federal agencies on matters bearing on problems of rations and control. Should not all members be able to use their professional equipment in intimate relation with the Army food problems either directly or through Selective Service channels?

In two wars we have seen the commercial rise and fall of dehydration as applied to fruits and vegetables and some other products, yet we fully know that this process of food preservation is rich in possibilities, and was of enormous value in the war. We have seen what were apparently properly prepared fruits and vegetables undergo changes in color and flavor by long storage under severe tropical conditions. We should be able to find a remedy by research. Other changes in well-prepared foods are known, especially in foods that we have used for defense and for supplying to less fortunate nations abroad. We are now investigating new methods of food preservation by high frequency and in other ways. In these we may find new procedures especially adapted to practical application in the quest for foods for defense, if unhappily, we are required to limit research in food production to this purpose. With the deep interest in food research and in new processes to broaden our food range on the home front, we should ever be on the alert to see wherein our new advances may find application also in the military or defense area.

The Committee recently established by the National Research Council undoubtedly has some of these broad objectives in view. Possibly a cooperating research committee from the I. F. T. could share in its work if coordinated assistance is sought.

Whether this be the case or not, we shall all join heartily in wishing it success in its efforts.

S. C. Prescott
President of I. F. T., 1939-1941

Editor's Note: The editorial on this page is the first in a series by past presidents of the Institute of Food Technologists, bearing on the relation of food technology to economic, health, and political problems of world-wide interest and importance. The second of the series will appear in the February issue.

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The Effect of Ultraviolet Light in the Meat Cooler on the Keeping Quality of Frozen Pork[a]

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When pork fat is exposed to ultraviolet radiation it is more susceptible to rancidity development during subsequent storage in the frozen state. Studies indicate that pork may have an appreciably shorter storage life at 0° or 10°F. when the meat cooler is equipped with ultraviolet ray lamps. The problem may be minimized by avoiding skinning the carcass or pork cuts prior to chilling or holding and by keeping the time between slaughter and freezing within 48 hours.

Introduction

The use of ultraviolet light in meat aging rooms has been widely accepted by the frozen food locker industry as a means of minimizing microbial growth and the development of off-odors. In beef aging rooms the use of ultraviolet light has made possible the maintenance of a higher room temperature to obtain a more rapid tenderization of the meat *(1, 4, 6)*. Freezer locker operators have been confronted with the problem of maintaining a sanitary plant, and of reducing contamination of the meat by microorganisms. The meat which is brought into the locker plant has been slaughtered and handled under a variety of conditions, which occasionally are very poor. Because of this situation many locker plant operators have installed ultraviolet lights in the meat aging rooms as a protective measure in reducing off odors and the development of slime and mold growth on the meat. A recent survey *(5)* indicates that 72 per cent of those frozen food locker plants in New York state having a meat cooler now are equipped with ultraviolet ray lamps.

Ozone is produced by the action of ultraviolet light on air. Whether the decrease in odors in aging rooms is the result of the ozone oxidizing the odorous gases or is due to a temporary blocking of the sense of smell is debatable. However, there is ample evidence that microbial growth can be significantly reduced by proper use of these lamps *(2, 3)*.

Both ultraviolet light and ozone are known to accelerate fat rancidity. This study was designed to determine to what extent UV lamps may affect the keeping quality, during prolonged frozen storage, of various meat products exposed to UV and ozone in the locker plant cooler.

Experimental

The keeping quality of meat may in part be expressed in terms of its tendency toward fat rancidification. The peroxide content of the fat before and during storage was used as a quantitative measure of the oxidation of fat in the meat. The fat was prepared for analysis by a cold extraction in low-boiling petroleum ether. The peroxide values (reported as m. eq. of peroxygen per kg. of fat) were determined by an iodometric procedure *(7)*.

A pilot study using beef, pork, and chicken was carried out to determine whether the use of ultraviolet lights in the cooler would shorten the life of these products in frozen storage. The beef loin was aged for ten days, the pork and poultry were held five days before packaging and storing at 0° F. The meat products indicated in Table 1 as "shaded UV treatment in cooler" were protected from the ultraviolet rays by means of a brown paper shield. The control samples were aged in a separate cooler not equipped with UV lights. The results of this exploratory study are summarized in Table 1.[c]

No detectable increase in peroxides in beef fat occurred during seven months' freezer storage even when ultraviolet ray lamps were operating in the cooler during the 10-day aging period. Chicken back fat did not become rancid after nine months' storage, whereas the fat taken from the abdomen, due to its exposure to air, had extremely high peroxide values after nine months. Again, the ozone or ultra-violet rays from the lamps had no effect on the storage life. The pork presented an entirely different picture in the exploratory study. The irradiated skinned pork loins showed a definite increase in peroxides during storage. The skin on the unskinned loin or shading the loin from the UV rays seemed to protect the fat against peroxide formation.

In this preliminary study, the samples were exposed to ultraviolet light in the meat cooler of a locker plant which had been using the lamps for some months, during which time the tubes had not been replaced or cleaned. Furthermore, only six lamps were installed in the rather large cooler. Accordingly, the UV irradiation was undoubtedly far below that recommended by manufacturers of the lamps.

In light of these results an experiment was planned wherein the studies on pork were expanded to give a more complete answer to the problem of chilling under ultraviolet light and the subsequent effects during storage.

Plan of experiment. The pork samples were subjected to the following conditions:

(a) One-half of the pork loins were skinned before being held in the cooler and the other half were held with the skin on to simulate the two procedures commonly employed in hog slaughtering and chilling.

[a] This study was supported in part by a grant by the Cooperative G. L. F. Exchange for meats research, a fellowship through the Philco Fund, and frozen food research grants by the Buffalo Niagara Electric Power Corporation; Carrier Corporation; Central Hudson Gas and Electric Corporation; Central New York Power Corporation; Consolidated Edison Company of New York, Inc.; Long Island Lighting Company; New York Power and Light Corporation; New York State Electric and Gas Corporation; Rochester Gas and Electric Corporation; and the Staten Island Edison Corporation.

[b] Philco Fellow.

[c] For many of the data in Table 1 we are indebted to Mr. L. H. Blakeslee, who initiated these early experiments.

TABLE 1

The effect of ultraviolet irradiation during aging on peroxide formation in beef, pork, and chicken fat during frozen storage

Product	Treatment in cooler	Initial	After treatment in cooler	(Months in storage at 0° F.)			
				4½	7	8	9
		1 [1] 2	1 2	1	1 2	1 2	2
				Peroxide Value [2]			
Beef	Control	2.0	1.9	....	0.4	....	....
	UV irradiated	2.1	1.6	....	0.6	....	....
	Shaded UV	2.1	5.0	....	0.7	....	....
Pork, skinned	Control	1.5-1.9	0.4-1.8	1.8	....	1.3-0.8	....
	UV irradiated	1.4-1.2	7.2-5.9	12.9	....	18.8-17.4	....
	Shaded UV	1.4-1.2	3.2-2.0	5.7	....	1.9-3.1	....
Pork, unskinned	Control	1.9-1.8	1.8-1.5	2.2	....	3.0	....
	UV irradiated	2.1-1.9	2.2-2.9	4.8	....	3.1-3.9	....
	Shaded UV	2.1-1.9	2.6-1.8	1.2	....	2.5-5.3	....
Chicken, back fat	Control	....	2.6-2.5	....	.... 4.0	....	7.7
	UV irradiated	....	1.4-3.2	....	.... 1.3	....	3.8
	Shaded UV	....	.... 1.9	....	.... 2.1	....	7.9
Chicken, abdomen fat	Control	1.8-2.5	2.7-1.7	....	.... 3.1	....	156.5
	UV irradiated	1.1-1.3	4.9-3.5	....	.... 5.7	....	176.8
	Shaded UV	0-1.1	1.5-2.4	....	.... 5.5	....	94.8

[1] The numbers 1 and 2 refer to two different samples. The peroxide values under heading 1 were all determined on the same sample. This is true also for the peroxide values listed under heading 2.
[2] Milliequivalents peroxygen/kg. of fat.

(b) The loins were held for two, four, or eight days in the meat cooler at approximately 36° F. and 90 per cent relative humidity. These prefreezing periods are not uncommon in locker plant practice.

(c) The upper sections of the loins were exposed to ultraviolet light (2537A) while the lower parts were shaded from the rays of the lamps to separately assess the effect of ozone generated by the lamps. Irradiation was provided by eight new Westinghouse Sterilamps, in a cooler 11 x 19 ft.[4] The intensity of UV output of the lamp nearest the meat (2 ft.) was 6.3 microwatts per sq. cm. per sec. at 38° F., the irradiation being measured at the source by means of a Rentschler "Click-Meter."

(d) All samples were frozen at 0° F. and stored at either 0° F. or 10° F.

Holding period. The pork carcasses with skin on were hung in the cooler for twenty-four hours after slaughter. The carcasses were removed from the cooler and the loins cut out. The loins were then hung at 36° F., either skinned or with the skin remaining intact. All control samples were obtained before the ultraviolet lights were turned on. The experimental samples were handled in this same manner, except that a shield of brown wrapping paper protected the bottom half of each loin from the UV light. This shield was constructed large enough to enable ample air circulation between it and the loin. The shield was allowed to hang approximately one inch below the loin to avoid interference from reflected ultraviolet light. Shielded samples were thus exposed to the ozone but not to the rays from the lamps.

At the end of the respective holding periods, the loins were removed from the cooler, the skin was removed from the previously unskinned loins, and the meat was cut into chops approximately one inch in thickness.

[4] We are indebted to the Empire Ultraviolet Equipment Co. for providing the Sterilamps.

Packaging, freezing, and storage. The individual chops were wrapped in one thickness of vaporproof Sylvania cellophane (P. M. F. B6 C. S., gauge 300) and then butcher-wrapped in a layer of medium weight brown wrapping paper. All samples were frozen for twenty-four hours at 0° F. After freezing, the samples were divided between storage compartments held at 0° and 10° F. Initial peroxide values were determined on all samples immediately after the freezing period. Storage samples were removed at appropriate intervals extending over a period of a year.

Sampling. The fat sample to be analyzed was obtained by cutting two thin strips of fat approximately 4 inches long and 1 mm. thick from the fatty edge of the chop, using a vegetable peeler. The use of this thin uniform outer strip of fat for analysis enabled an early detection of rancidity, since both the irradiation and the oxygen penetrate only a few millimeters. This method of sampling the fat is reproducible to within a few percent, and provides a 3-5 gram sample for analysis.

The fat was immediately extracted by grinding with purified sand and low-boiling petroleum ether. Anhydrous sodium sulfate was added during the grinding to remove the water present in the fat. This mixture was filtered into a 50 ml. volumetric flask and made to volume. The petroleum ether extracts could be held one or two days at 38° F. without causing any change in the peroxide value. The peroxide values were determined by the method referred to earlier *(7)*.

Results

The experimental data from this holding and storage study on pork samples are given in Tables 2, 3, and 4. The control samples, those held in the cooler without exposure to ultraviolet light or the traces of ozone produced, showed no significant formation of fat peroxides during one year's storage at 0° F. At 10° F. several of the samples were close to incipient rancidity (per. value = 15-20) at 44 weeks (Table 2).

The formation of peroxides in the experimental *unskinned* pork samples was similar to that in the control samples. The skin of the carcass protected the fat from any deleterious action of ozone, air, or of the ultraviolet light.

Removal of the skin before storing the meat in the freezer thus eliminated the exposed surface in which peroxide formation might have been accelerated. After 44 weeks' storage at 0° F. none of the samples showed incipient rancidity, and after 52 weeks only one had exceeded it (Table 3). Again, storage at 10° F. accelerated the formation of peroxides in the fat as indicated by elevated peroxide values at the 44- and 52-week sampling periods.

The formation of peroxides in the irradiated *skinned* pork samples was very rapid. The values for samples held four and eight days were approaching or had already passed the point of incipient rancidity when the initial analyses were made before placing the samples in freezer storage. On the other hand, the shaded skinned pork samples stored at 0° F. did not show an increase in peroxide value even after one year's storage (Table 4). Only one of three shaded skinned samples held at 10° for a year (that which had been held 8 days prior to freezing) had passed the point of incipient rancidity.

This apparent stability of the pork fat in samples exposed to conditions generally considered to be harmful (several days' delay in processing; storage at 10° rather than the recommended 0° F. or lower), was rather unexpected, as was the inconsistency of peroxide values of several other samples stored at 10° F. It was observed that at this storage temperature the pork fat developed rancid off odors and yet the peroxide values remained quite low (Tables 2, 3, 4). Several pork samples which had been stored at 10° F. and which showed unexpectedly low peroxide values were held for two weeks in the 0° F. freezer, then the peroxide values were repeated. In nine out of eleven such repeat analyses, the peroxide values at the end of the additional two-week period showed increases to levels ranging from 1.5 to 10 times the peroxide value at the end of the 10° F. storage period. This would indicate that the peroxides had been decomposing at a rate similar to the rate of formation when the pork fat was stored at 10° F. The peroxides, which are unstable intermediate products in fat rancidification, accumulated, rather than decomposing, when the pork samples were transferred to the 0° F. freezer. These observations suggest that the use of the peroxide determination as an objective, quantitative means of evaluating the progress of rancidity in frozen pork, while quite acceptable at a storage temperature of 0° F., may not be suitable at elevated storage temperatures.

Several practical implications are evident from the results of the present study. (a) A storage temperature

TABLE 2

The formation of peroxides in the control pork samples during frozen storage at 0 and 10° F.

Animal Number	Holding Period, Days	Condition of Carcass	Storage Temperature, ° F.	Storage period in weeks: Initial	8	16	44	52
				Peroxide value [1]				
1	2	Skinned	0	0	2.3	1.3	0.9	2.1
2	4	Skinned	0	0	1.2	0.2	0.8	2.1
3	8	Skinned	0	0	0.8	0.7	0.6	2.8
7	2	Unskinned	0	0	1.8	1.3	0	2.6
8	4	Unskinned	0	0	0.5	0	1.1	1.4
9	8	Unskinned	0	0	0.8	1.6	2.2	2.8
2	2	Skinned	10	0	0.7	0.7	13.8	10.2
3	4	Skinned	10	0	1.0	0.7	3.4	5.4
1	8	Skinned	10	0	2.0	0.8	2.1	2.9
8	2	Unskinned	10	0	0.7	0.4	16.0	43.0
9	4	Unskinned	10	0	1.1	0	2.5	3.9
7	8	Unskinned	10	0	1.7	1.7	10.1	40.2

[1] Milliequivalents peroxygen/kg. of fat.

TABLE 3

The formation of peroxides in the experimental unskinned pork samples during frozen storage at 0 and 10° F.

Animal Number	Holding Period, Days	Irradiated or Shaded	Storage Temperature, ° F.	Storage period in weeks: Initial	4	8	12	24	32	44	52
				Peroxide Value [1]							
10	2	Irradiated	0	3.1	0	0	1.3	0	....	0.6	0.7
11	4	Irradiated	0	0	0	1.2	1.6	1.4	....	2.2	5.9
12	8	Irradiated	0	1.9	1.9	2.2	0.8	1.3	....	1.2	4.4
10	2	Shaded	0	0	0	0	1.1	1.5	....	0.4	1.7
11	4	Shaded	0	0	0	0.9	2.6	0	....	5.7	6.8
12	8	Shaded	0	2.8	1.5	2.2	1.6	0	....	5.0	28.9
11	2	Irradiated	10	1.2	0.7	1.0	....	2.6	1.4	3.2	1.5
12	4	Irradiated	10	0	1.5	0.8	....	2.4	1.8	23.7	33.7
10	8	Irradiated	10	0	2.0	1.7	....	3.0	4.5	9.4	1.9
11	2	Shaded	10	0.6	0	0.9	....	1.6	1.7	10.5	18.3
12	4	Shaded	10	0	2.0	0.5	....	4.0	6.0	3.8	21.7
10	8	Shaded	10	0	2.0	2.1	....	2.6	2.0	2.0	2.3

[1] Milliequivalents peroxygen/kg. of fat.

TABLE 4

The formation of peroxides in the experimental skinned pork samples during frozen storage at 0 and 10° F.

Animal Number	Holding Period, Days	Irradiated or Shaded	Storage Temperature, ° F.	Storage period in weeks								
				Initial	4	8	12	20	24	32	44	52
				Peroxide Value [1]								
4	2	Irradiated	0	2.0	6.7	5.0	10.3	....	27.3	....	....	....
5	4	Irradiated	0	9.3	14.0	13.3	13.3	....	9.0	....	....	....
6	8	Irradiated	0	37.0	38.0	65.3	45.2	....	95.2	....	....	....
4	2	Shaded	0	0	0	0	1.4	0.5	....	....	....	....
5	4	Shaded	0	0	0.7	1.1	....	....	....	0.6	0.5	9.6
6	8	Shaded	0	1.3	2.2	2.8	....	3.0	....	2.2	1.1	3.0
5	2	Irradiated	10	6.5	9.0	8.9	9.6	7.1	5.3	....	....	....
6	4	Irradiated	10	18.4	22.4	27.8	32.1	....	48.3	....	....	....
4	8	Irradiated	10	23.1	30.4	51.5	38.7	....	81.3	....	....	....
5	2	Shaded	10	0	0	0.7	....	....	....	2.3	5.7	2.9
6	4	Shaded	10	1.0	1.9	2.3	....	1.1	....	2.2	2.8	2.5
4	8	Shaded	10	1.6	1.3	3.0	....	1.6	....	4.8	13.1	26.0

[1] Milliequivalents peroxygen/kg. of fat.

of 10° F. is too high for prolonged storage of frozen pork. (b) If ultraviolet lights are to be used in the meat cooler, the practice of leaving the skin on hog carcasses until immediately prior to processing is strongly to be recommended. (c) The small amount of ozone generated by ultraviolet lamps at 2537A has a negligible effect on the stability of the fat. This confirms the observations of Fredholm *(3)*. (d) A few days' delay in processing pork hung in coolers equipped with ultraviolet lamps may greatly shorten the storage life of the frozen product.

Summary

Ultraviolet irradiation of skinned pork carcasses during the period prior to freezing proved undesirable if the holding period exceeded two days. The fat of such meat turned rancid much more rapidly during subsequent freezer storage.

Shading the pork carcasses from the ultraviolet irradiation prevented the rapid formation of peroxides in the surface layers of fat, indicating that ozone played no important role in the rancidification catalyzed by the ultraviolet ray lamps.

Holding the carcasses with the skin on, and then removing the skin before storage, protected the pork fat against the deleterious action of the ultraviolet light.

Storage of pork at 10° F. accelerated the formation of peroxides in the pork fat and also the development of rancid odors. This elevated storage temperature particularly affected the stability of the irradiated pork samples.

Inconsistencies in the trend of peroxide values of pork fat stored at 10° F. emphasized the need for a method of evaluating the end products of fat oxidation rather than the determination of the unstable intermediate peroxides.

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If you have the qualifications for full membership in the Institute a change of status to full Member is easily accomplished as follows:

1. Be reasonably certain that you are qualified for full membership.

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2. Write to the National Secretary or to the Secretary of your Regional Section for a "Change of Status" application form.

3. Fill this out with the exception of the signatures of three sponsors, and proceed according to the instructions for handling an application for membership on page 89 of the April 1948 issue of Food Technology.

Use of Liquid Sugars in Freezing of Apricots, Peaches and Nectarines[a]

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Data are presented to show the effect of various sugars on quality of frozen apricots, peaches, and nectarines. Sugar absorption, rate of auto-oxidation of ascorbic acid and rate of hydrolytic transformation of dehydro-ascorbic acid are shown as some of the basic data required to evaluate the protective effect of sugar for fruit.

Although a considerable amount of data on the most suitable procedures for freezing fruits in sirup to obtain maximum retention of color and flavor has been accumulated since the introduction of the use of sirup for frozen fruits in 1920 *(3)*, most of this data is based largely on subjective examination of experimental packs. There is relatively little technological data on the basis of which proper selection of sugar for use in freezing of fruit products could be made, and only rarely have experimental packs been made under commercial conditions from the packing lines *(5)*. In the evaluation of the most desirable types of sugars and densities of sirups to be used in the preparation of fruit for freezing storage, and of the best methods of using these sirups, it is necessary to know the ease with which they spread over fruit tissues, are held or are absorbed by them; their effect on rate of cooling, ice formation, and separation of unattractive sugar eutectics; their effect in preventing or minimizing changes in color brought about either by changes in natural pigments or discoloration by enzymic or non-enzymic oxidative processes; their effect on flavor, both as regards compatability with natural fruit flavors and their ability to promote the retention of the original flavors; and their effect on texture particularly from the colloidal point of view *(5)*.

To obtain technological data of this type, an extensive investigation of the freezing of apricots, peaches and nectarines was begun in our laboratories in the summer of 1946. Experimental packs were made to study the effect of various methods of applying sirup; to compare sucrose-sirups with those containing 30, 50, 70 and 90 percent invert sirup, and with those prepared from corn sugar, low conversion and high conversion corn sirup; and to compare the effectiveness of several anti-oxidants both with and without added citric acid. Fruit from the same source was used in all lots of sixteen packages each, and it was packed under commercial conditions in heat-sealed cellophane bags in top-opening Menasha cartons, overwrapped with waxed paper, frozen in a 14-stage Finnegan cross draft freezer, and stored at 0° F. Fifty-two lots of firm-ripe Blenheim apricot samples were packed on July 24 and 25, 1946, and graded November 13-17, 1946, April 2-7, 1947, and September 8-11, 1947. Forty-nine lots of Rio-Oso-Gem peaches were packed on August 14, 1946, and graded as above; nineteen lots of Kim and twenty-eight lots of Gower were packed on July 24 and 25, 1946, and graded the above dates. The samples were graded by about six tasters for color, flavor, texture and odor immediately after defrosting. In addition the drained weight of fruit, weight and density of cut-out sirup, color (by comparison with color charts and by photo-electric-colorimetry of alcoholic extracts), tannin content, ascorbic acid content, sugar content, acidity and pH of drained fruit and cut-out sirup were determined.

The solubility of oxygen in solutions of various sugars, and the effect of various sugars on the rate of oxidation of ascorbic acid, and on the rate of transformation of the primary product of oxidation, dehydro-ascorbic acid into 2, 3 keto gulonic acid, was measured.

This report will be limited to the presentation of data on the comparison of invert sirups with sucrose sirups, because of the present trend in the industry toward labor saving by the bulk handling of sugar. The distribution of solutions of sucrose, and of partially or almost completely inverted sucrose solutions, has been of great value to large users of sugar in reducing handling and storage costs and in facilitating preparation of sirups for use in various products *(4, 18)*. Although the more common physical properties of solutions of sucrose and of mixtures of sucrose and invert sugar have been summarized *(10)*, the possibilities and limitations of their use in the freezing of fruit products were not determined previously. Our data on this point are presented here together with preliminary data on sugar absorption, oxygen solubility and ascorbic acid transformation.

Acceptability of Invert Sirup in Frozen Fruit

Firm, ripe Blenheim apricots grown in Hollister, washed, halved and pitted, were taken from the packing line after sorting. Ten ounces of the fruit were placed in a cellophane bag in a one-pound top-opening Menasha carton, covered with 200 ml. of a 40° Brix sirup containing varying amounts of invert sirup, sealed, overwrapped, and frozen in 4 to 7 hours in a 14-stage Finnegan freezer at an air temperature of about —40° F. They were then stored at 0° F. until tested. Firm, ripe Rio Oso Gem peaches from Hollister, washed, steam-peeled, and sliced, were packed in similar cartons at the rate of 11 ounces of fruit to 5 ounces of 40° Brix sirup. Kim nectarines, a highly colored, yellow-fleshed, freestone nectarine, and Gower nectarines, a highly colored, white-fleshed, freestone nectarine, were washed, halved, pitted and packed at the rate of 10 ounces of fruit to 8 ounces of 40° Brix sirup in similar cartons, frozen and stored at 0° F. The nectarines were sorted for maturity so that the samples packed were from lots of fairly uniform quality, but the apricots and peaches were taken from the packing lines so that there was the same degree of variation in maturity that occurred in commercial samples. The analysis of composite samples

[a] Presented before the Eighth Annual I. F. T. Convention, Philadelphia, Pa., June 9, 1948.

of fresh fruit and of fruit preserved by dropping quartered or sliced fruit into hot 95 percent alcohol and heating for 30 minutes to inactivate invertase and other enzymes is shown in Table 1.

TABLE 1

Analysis of Composite Samples, Fresh and Preserved in Alcohol

	Apricots	Peaches	Kim Nectarines	Gower Nectarines
Preserved Samples				
Total solids	16.25	12.37	16.72	20.41
Alcohol soluble solids	14.00	10.67	12.60	17.73
Water insoluble solids	1.45	1.16	1.41	1.82
Acidity as citric	1.09	0.75	.....	0.90
Sucrose	6.69	4.06	6.66	8.23
Reducing sugars as glucose	2.75	3.37	2.40	3.05
Tannins	0.049	0.114	0.056	0.068
Fresh Samples				
Acidity	0.91-1.18	0.81	0.76	0.72
pH	3.75-4.0	3.67	3.80	4.13
Total soluble solids	14.50	11.70	17.10	20.10

Samples of the frozen apricots were removed from storage after about 3½ months, 8 months, and 12½ months; and 3, 8, and 13 months for peaches and nectarines. These were allowed to defrost at room temperature for about 5-6 hours while standing on edge in the carton, then opened and spread out on cut-out trays. The samples were observed for color immediately after all the ice had melted, and were tasted for flavor and texture while still cool (about 50° F.). The tastings were made on groups of twelve to fifteen samples, since we were interested in detecting readily noticeable differences in color, flavor and texture, rather than in discerning small differences. At each tasting, one lot was graded by all the tasters together and then used for comparison with the others. In judging the samples a numerical score was used: 1—Poor, 2—Fair, 3—Good, 4—Very Good, 5—Excellent. The taste panel was composed of from six to eight judges, three or more from our laboratory, and three from the C and H Sugar Refinery. Only two of the tasters were experienced judges of frozen fruits. The average scores obtained are shown in Tables 2 to 4.

TABLE 2

Subjective Scores for Apricots in 40° Bal. Sucrose and Invert Sirups

Code	%	Color			Flavor			Texture		
	Invert	I	II	III	I	II	III	I	II	III
AIA	0	2.7	2.4	2.5	2.9	2.9	2.0	3.0	2.9	2.0
AIIA-1	30	1.9	2.4	1.6	2.6	2.6	1.4	3.4	2.7	1.9
AIIA-2	50	2.6	2.8	1.6	2.8	3.0	1.5	3.2	2.6	1.8
AIIA-3	70	2.3	2.8	1.6	2.6	2.7	1.6	2.2	2.8	1.8
AIIA-4	90	2.6	2.7	1.8	2.6	2.5	1.8	3.3	2.9	1.8
AIIIA-3	0	2.5	2.9	2.3	3.0	2.7	2.7	3.0	2.7	2.7
AIIIA-9	90	2.9	2.2	2.0	3.0	2.7	2.0	2.8	2.6	2.8

TABLE 3

Subjective Grades for Peaches in 40° Bal. Sucrose and Invert Sirups

Code	%	Color			Flavor			Texture		
	Invert	I	II	III	I	II	III	I	II	III
P-3	0	3.0	2.1	2.2	3.0	2.3	2.0	3.5	2.5	3.0
P-6	30	2.6	2.1	1.8	2.9	2.9	2.6	3.4	2.8	2.7
P-7	50	3.1	2.1	2.3	2.9	2.4	2.4	3.3	2.6	2.8
P-8	70	3.1	1.6	1.9	2.7	2.6	1.5	3.3	2.5	2.5
P-9	90	2.6	1.8	1.8	2.6	2.4	1.4	2.9	2.8	2.9
P-35	0	2.7	2.1	1.9	3.2	2.1	1.4	3.0	2.7	2.4
P-45	0	2.6	2.0	2.0	3.1	2.3	2.1	3.5	2.4	2.5
P-41	90	2.5	1.8	2.0	2.8	1.7	1.3	3.1	2.3	2.0

TABLE 4

Subjective Grades for Nectarines in 40° Bal. Sucrose and Invert Sirups

Code	%	Color			Flavor			Texture		
	Invert	I	II	III	I	II	III	I	II	III
NK-2	0	3.4	3.4	3.0	2.9	2.9	3.0	3.0	3.3	3.0
NK-3	30	2.9	3.4	2.9	2.9	3.2	2.8	2.9	3.2	2.6
NK-4	50	3.1	3.6	2.9	3.2	3.2	2.8	2.9	2.9	2.9
NK-5	70	3.2	3.4	2.7	2.4	2.9	2.8	3.2	3.4	2.7
NK-6	90	2.7	3.1	2.6	3.0	3.0	2.7	3.0	3.2	2.8
NG-2	0	2.7	3.0	3.0	3.0	3.0	2.9	3.2	3.0	2.4
NG-10	90	2.9	2.6	3.0	3.4	2.8	3.0	3.4	2.8	3.0

The average scores for apricots indicate relatively little difference in appearance between apricots frozen in sucrose sirups and those frozen in invert sirups at the first and second tasting, but a definite decrease in score due to progressive discoloration in all invert sirups was apparent at the last tasting. There was no definite trend in texture, probably owing to variability of samples, except a marked increase in relative toughening of the skin with increase in storage period. This is reflected in a decrease of from 1 to 1.5 points in score, and this decrease was more noticeable in the samples packed in invert sirups. In flavor, at the first tasting, there was a noticeable difference between the 70 percent invert sirups and those of lower degree of inversion. This difference persisted in subsequent tasting. There was a noticeable decrease in flavor retention with storage time, the decrease being greater in apricots packed in invert sirups than those packed in sucrose sirups. The more discerning tasters detected an appreciable off-flavor at 70 percent inversion and a marked off-flavor at 90 percent.

In peaches there was little readily detectable difference in color between fruit frozen in sucrose sirups and those frozen in invert sirups at the first tasting, except at 90 percent inversion. The color score decreased with increase in storage time, the decrease being most noticeable at 8 months, but it was similar in sucrose and invert sirups. There was a noticeable decrease in texture at 90 percent inversion, but the trend was not definite owing to variations within the samples. As in the case with apricots, the texture of the samples was appreciably poorer with increase in storage time. There was an appreciable change in flavor at 70 percent inversion and a noticeable one at 90 percent which was more marked to the more discerning tasters. The flavor of the peaches decreased with increase in storage time, and this decrease was greater in the peaches frozen in 70 percent and 90 percent invert sirup. It was particularly noticeable as a combination of oxidized and foreign flavor, to the discerning tasters.

In Kim nectarines there was an appreciable discoloration at 90 percent inversion which persisted throughout the storage period. Kim nectarines retained their color better, however, than did apricots or peaches. In texture there was little difference between the sucrose and invert sirup samples. Unlike apricots, the nectarine skin was thinner and tender, and remained so during storage. In flavor there was a slight difference at first in invert sirup, noticeable at 70 percent inversion, but this difference did not persist, owing largely to variability of samples. In general, nectarines were of higher flavor initially, and retained their color and flavor better

TABLE 5

Distribution of Soluble Solids After 6-8 Months Storage at 0° C.

Code	Ingoing Weights Grams		Drained Weights Grams		Soluble Solids %		pH		Acidity % as citric	
	Fruit	Sirup	Fruit	Sirup	Fruit	Sirup	Fruit	Sirup	Fruit	Sirup
AIA	280	235	201	242	17.9	35.6	3.85	3.91	0.69	0.32
AIIA-1	280	235	282	238	17.8	34.0	3.80	3.87	0.60	0.39
AIIA-2	280	235	251	244	18.4	32.4	3.80	3.85	0.53	0.46
AIIA-3	280	235	252	248	18.9	30.1	3.75	3.80	0.65	0.46
AIIA-4	280	235	258	248	19.9	31.6	3.80	3.80	0.63	0.44
AIIIA-3	280	235	282	218	17.1	37.0	3.34	3.49	0.70	0.32
AIIIA-4	280	235	273	242	17.1	43.1	3.22	3.34	0.70	0.40
P-3	312	142	294	149	19.0	21.9	3.70	3.67	0.53	0.47
P-6	312	142	289	152	19.0	23.0	3.69	3.72	0.50	0.55
P-7	312	142	296	145	19.0	24.0	3.69	3.70	.....	0.44
P-8	312	142	274	163	21.4	21.0	3.79	3.90	0.46	0.47
P-9	312	142	272	145	19.8	21.0	3.71	3.85	0.53	0.51
P-35	312	142	315	123	17.5	23.7	3.68	3.72	0.49	0.49
P-45	312	142	295	140	18.0	25.4	3.62	3.64	0.46	0.47
P-41	312	142	289	108	19.2	23.2	3.61	3.71	0.51	0.46
NK-2	284	227	267	243	22.9	32.9	3.86	4.13	0.35	0.16
NK-3	284	227	271	234	21.7	32.7	3.84	4.20	0.50	0.20
NK-4	284	227	248	253	22.6	31.4	3.50	3.70	0.46	0.33
NK-5	284	227	268	228	21.9	32.9	3.79	4.00	0.58	0.24
NK-6	284	227	261	238	24.4	32.1	3.99	4.17	0.44	0.23
NG-2	284	227	261	240	23.5	34.2	3.75	3.90	0.62	0.27
NG-10	284	227	255	161	25.4	29.3	3.92	4.02	0.50	0.37

during freezing storage than did the apricots or peaches. In Gower nectarines there was no appreciable difference in color, flavor, or texture between sucrose and 90 percent invert sirup samples. The more careful tasters reported less difference between nectarines frozen in sucrose and in invert sirups than was found for apricots and peaches.

The analytical data obtained on these samples are summarized in Tables 5 to 7. Since these data were obtained by several individuals whose laboratory technique differed, they are subject to variable analytical errors, and in addition are subject to sampling errors caused by the purposely uncontrolled variation in composition with the normal variation in maturity occurring under commercial processing conditions. Again our attention was focused on significantly large changes and trends, rather than on small differences.

The data summarized in Table 5 indicate no significant trend in drained weight with degree of inversion of sirup and little change in soluble solids, pH, or acidity. In comparison with similar determinations on fresh fruits, the soluble solids content of frozen apricots increased by about 4 percent, the pH of the fruit increased, and the acidity decreased, largely due to movement of acids out of the fruit tissue into the sirup. These changes occurred largely during defrosting. The soluble solids content of peaches increased by about 6 percent, and of nectarines by about 4 percent. The peaches remained at about the same pH value after freezing and freezing storage and defrosting, but their acidity decreased. The pH values of Kim nectarines were essentially the same in fresh and frozen fruit, but those of Gower nectarines decreased slightly. The acidity in fruit tissues decreased apparently, owing to leaching.

The data in Table 6 show a comparison between the color rating and actual color as shown by comparison with Maerz and Paul color charts *(12)*. There was no significant change in tannin content of the frozen apricots and nectarines, but a definite decrease in that of peaches, partly due to leaching and partly to oxidation. There was an appreciable increase in sugar content, but very little significant change in sucrose content of the samples packed in sucrose and invert sirup. The available data were too erratic for definite conclusions other than that the natural sucrose content was not appreciably altered by invertase activity.

The degree of retention of the naturally present ascorbic acid content of the fruit samples is shown in Table 7 after 4-5 months and 11-12 months of storage. The original ascorbic acid content of the fresh fruit was not determined. The more commonly accepted values for the ascorbic acid content of apricots vary from 4-10 mg. per 100 gm. and of nectarines 8-25 per 100 gms. The values we found for samples prepared under conditions such as to eliminate enzymic oxidation are in the same range, about 8 mg. for apricots and peaches and about 20 mg. for nectarines. In general the data indicate that the ascorbic acid retention in apricots and peaches was much poorer than in nectarines, particularly the Gower

TABLE 6

Color Rating, Tannin and Sugar Content

Code	Color Rating	M & P Color	Tannin Content		Sugars		
			Fruit	Sirup	Soluble Solids as Sugars	Sucrose	Total
AIA	2.4	P10J10	0.011	0.019	17.9	11.1	14.5
AIIA-1	2.4	P11J11	0.065	0.025	17.8	9.3	14.7
AIIA-2	2.8	P11K9	0.077	0.029	18.4	6.2	12.8
AIIA-3	2.8	P10L9	0.020	0.062	18.9	6.3	14.5
AIIA-4	2.7	P11K9	0.005	0.038	19.9	5.1	14.5
AIIIA-3	2.9	P10I9	0.049	0.048	17.1	7.6	10.9
AIIIA-4	2.5	P11K10	0.049	0.055	17.1	9.1	12.2
P-3	2.1	P11K7	0.084	0.046	18.1	11.2	15.5
P-6	2.1	P11K7	0.049	0.042	19.1	6.7	14.1
P-7	2.1	P11K7	0.029	0.029	.....	.....	.....
P-8	1.6	P12K8	0.029	0.000	21.4	.....	14.4
P-9	1.8	P13J8	0.031	0.017	.....	.....	.....
P-35	1.9	P11L8	0.054	0.023	17.5	8.1	10.8
P-45	2.0	P11L8	0.071	0.013	.....	.....	.....
P-41	2.0	P11K8	0.041	0.035	19.2	4.1	16.4
NK-2	3.4	P11L10	0.033	.....	22.9	.....	.....
NK-3	3.4	P11L9	.....	.....	21.7	5.0	9.4
NK-4	3.6	P11K10	.....	.....	22.6	14.6	19.5
NK-5	3.4	P10J8	.....	.....	.....	.....	.....
NK-6	3.1	P11K9	.....	.....	.....	.....	.....
NG-2	3.0	P5H7	0.052	0.013	23.5	16.0	18.7
NG-10	3.0	P6F10	0.065	0.000	.....	.....	.....

TABLE 7

Ascorbic Acid Content in Mg. per 100 grams

Code	After 4-5 months				After 11-12 months			
	Drained Weight		Ascorbic Acid		Drained Weight		Ascorbic Acid	
	Fruit	Sirup	Fruit	Sirup	Fruit	Sirup	Fruit	Sirup
AIA	256	214	0.9	0.7	245	228	4.0	0.0
AIIA-1	267	195	1.5	0.0	248	230	3.2	0.22
AIIA-2	271	217	0.0	0.0	256	238	0.45	0.21
AIIA-3	273	210	0.0	0.0	259	195	0.58	0.65
AIIA-4	270	224	0.0	0.0	255	235	0.0	0.0
AIIIA-3	270	217	0.1	0.2	259	207	0.0	1.05
AIIIA-4	265	137	2.6	0.7	256	154	1.2	0.26
P-3	329	71	1.5	1.0	302	106	0.93	0.0
P-6	299	103	1.6	0.5	314	90	0.32	0.0
P-7	305	103	1.4	0.6	293	113	0.51	1.9
P-8	306	111	1.7	0.4	317	91	0.47	0.0
P-9	301	121	0.9	0.3	306	102	0.33	0.0
P-35	299	124	1.3	0.0	288	127	0.70	0.2
P-45	293	121	0.8	0.0	283	126	0.31	0.12
P-41	303	123	0.4	0.0	293	114	0.17	0.0
NK-2	225	226	0.0	2.0	249	191	9.0	2.2
NK-3	256	230	0.0	2.6	246	222	9.4	2.6
NK-4	276	201	9.0	2.3	279	198	7.9	2.7
NK-5	271	214	6.7	1.7	260	215	8.0	2.0
NK-6	268	214	8.4	2.0	268	204	5.7	1.1
NG-2	288	143	17.4	6.5	279	192	23.6	8.7
NG-10	275	196	10.6	5.5	252	150	20.4	9.2

variety. There is some indication that in apricots ascorbic acid retention was progressively poorer in sirups of increasing degree of inversion. This trend was not observed in peaches, and Gower nectarines, but did occur in Kim nectarines. Other observations indicated that the phenolase oxidation of ascorbic acid was inhibited more by sucrose than invert sirup, but these investigations are still in progress. The ascorbic acid losses were greatest in the initial storage and did not increase with length of storage period; if at all, the recovery was better from samples frozen for one year than those frozen for four months. The sirups drained from the fruit contained little ascorbic acid except in the case of nectarines. In Kim nectarines ascorbic acid retention was much better in sirups containing 70 percent or less of invert sugar than in the 90 percent invert sirups.

Absorption of Sugar by Frozen Fruit

The absorption of sugar by fruit frozen with added sugar or sirup has long been considered desirable to improve preservation of color and flavor and to yield a better tasting product. To obtain better sirup penetration in berries, Wiegand *(20)* suggested cold storage of the packaged fruit and sirup at 30-31° F. for 24 to 72 hours, and this was confirmed by extended tests in our laboratories *(7)*. The total quantity of sugar absorbed by the fruit, however, is small in comparison with change in weight caused by withdrawal of water as shown in Table 8. In addition to delayed freezing, impregnation deaeration was investigated in our laboratories. Most of the sugar exchange occurred during defrosting rather than freezing storage in our studies which will be reported in more detail elsewhere.

Effect of Sugars on Oxygen Solubility

Since oxidation during freezing, freezing storage, and defrosting is detrimental to color and flavor retention, it is desirable to reduce this to a minimum at all stages of processing and distribution. The rate of this oxidation is determined by the amount of oxygen present in fruit tissues, the amount of oxygen present in the sirup added to the fruit, and the amount of oxygen diffusing from the surrounding air into the package. It has been commonly observed that in preparing sirups by dissolving granulated sugar in water at room temperature considerable quantities of oxygen are dissolved in the sirup. Repeated tests have shown large amounts of oxygen in cold sirups as used in plants. To obtain data on the solubility of oxygen in sugar solutions and the effect of concentration and method of production on oxygen content, the oxygen content of a number of sirups under various conditions was obtained by a modified polarographic method *(11)*. These data *(9)* were briefly summarized at the Annual Meeting of the Northern California Section of the Institute of Food Technologists on December 4, 1947, and are presented in part here. As the data in Table 9 indicate there is a marked reduction in solubility in sugar sirups in comparison with water, the solubility of oxygen being least in a 90 percent inverted sirup, and less in corn sirups than in sucrose solutions.

TABLE 9

Oxygen Solubility of Various Sugar Sirups at 40° Bal. and 22° C.

Sugar	Oxygen Solubility in ml. per 100 ml. of sirup
Control	0.636
Cane sugar	0.256
Corn sugar	0.238
Levulose	0.227
Dextrose	0.218
High conversion corn sirup A	0.204
High conversion corn sirup B	0.204
Invert sirup (0-90)	0.105

TABLE 8

Water Lost and Sugar Absorbed in 50° Bal. Sirup Packs

Fruit	Treatment	Water Lost %	Sugar Absorbed %	Authority
Strawberries	whole	39.35	2.75	Wiegand (21)
Strawberries	whole, frozen at once	42.5	3.2	Joslyn & Marsh (7)
Strawberries	whole, stored for 24 hrs. at 32° F.	40.0	6.7	Joslyn & Marsh (7)
Strawberries	whole, frozen at once	28.4	3.7	Joslyn & Marsh (7)
Youngberries	whole	22.6	0.0	Marsh (13)
Red raspberries	whole	22.2	5.25	Wiegand (21)
Boysenberries	whole	13.70	0.30	Wiegand (21)
Apricots	halved	5.35	3.19	Wiegand (21)
Apricots	sliced, frozen at once	24.2	5.1	Marsh (13)
Apricots	sliced, stored at 32° F. for 24 hrs.	29.0	4.0	Marsh (13)
Apricots	sliced, stored at 32° F. for 48 hrs.	30.2	3.1	Marsh (13)
Apricots	sliced, stored at 32° F. for 72 hrs.	30.8	5.6	Marsh (13)
Peaches	sliced	30.80	1.75	Wiegand (21)

The effect of method of preparing sirups is shown in Table 10. In one case the sirups were prepared by dissolving the necessary amount of granulated sugar in water at 20° C. with vigorous stirring; in the other case the water was brought to boiling and the sugar dissolved with minimum of stirring and then allowed to cool to 20° C. in uncovered beakers. There was little difference under these conditions at lower densities, but the percentage difference progressively increased with increase in density. Some diffusion of oxygen undoubtedly occurred during cooling of the hot sirup, but this would also occur under commercial practice. These data indicate but little difference between the two methods. Under commercial practice, however, occluded oxygen as well as dissolved oxygen would be incorporated in the sirup.

TABLE 10

Effect of Method of Dissolving Granulated Cane Sugar on Oxygen Content

	Sirup density, ° Bal.			
	20	30	40	50
Solution in cold with stirring	0.315	0.227	0.168	0.108
Solution in hot	0.305	0.219	0.142	0.078

Relative Effect of Sugars on the Rate of Auto-Oxidation of Ascorbic Acid

It has been demonstrated in our laboratory and elsewhere that darkening and discoloration of fruit tissue injured by freezing, mechanical injury, or by suboxidation will not occur until the residual ascorbic acid content is reduced below the level at which it will maintain the chromogens (phenolic derivatives) in the reduced stage. Thus it is important to conserve both the naturally occurring ascorbic acid and that added in processing. Oxidation of ascorbic acid, by both enzymic and non-enzymic processes, is inhibited to varying degrees by sugars, and we have found that sugars may exert a specific effect in both instances. Investigations on the extent and mechanism of this effect have been under way in our laboratory for the past two years as well as earlier *(7)*. That sugars do exercise a protective effect on non-enzymic oxidation of ascorbic acid has been established by others *(2, 14, 17, 19)*, but there is a considerable confusion in the literature on the relative effectiveness of the sugars.

Richardson and Mayfield *(17)* reported that sucrose and white corn sirup exerted a protective influence on ascorbic acid oxidation while dextrose showed a tendency toward destructive influence during boiling. Munilla and Vogelsinger *(14)* reported that addition of sugars reduced the rate of oxidation of ascorbic acid solutions stored at 20° C., lactose having least effect and dextrose and sucrose most. The Japanese investigations *(19)* reported that citric acid, fructose, and sucrose promoted oxidation while glucose inhibited it. Chamrai *(2)* established the protective effect of sucrose and pointed out that at low pH this was due to its effect on reducing rate of diffusion of oxygen into his solutions, and at higher pH values to its copper-binding power.

Our data indicate that this in part can be explained by differences in Cu and Fe content caused by the presence of variable amounts of these metals in the sugars used and by differences in oxygen tension. We have investigated the effectiveness of various sugars at various pH values and under two different experimental conditions on the auto-oxidation of ascorbic acid *(8)*.

TABLE 11

Effect of Sugars on Auto-Oxidation of Ascorbic Acid Solutions Allowed to Stand Exposed to Air at 22° C. in Presence of 40% Sugar or Sirup Solids

Sugar	Cu p.p.m.	Fe p.p.m.	Percent of ascorbic acid oxidized	
			24 hours	48 hours
None	.....	.....	57.2	83.5
Low conversion corn sirup	0.70	8.3	19.2	30.4
High conversion corn sirup B	1.13	28.1	10.0	14.7
High conversion corn sirup A	9.85	10.0	26.5	48.2
Dextrose	0.34	.....	12.4	42.5
Levulose	0.10	7.6	12.6	17.8
Lactose	0.70	14.4	17.3	30.1
Maltose	0.20	11.2	8.2	16.3
Sucrose	0.0	5.0	18.0	31.7
Sirup (type 0)	0.39	15.2	21.5	36.7
Sirup (type 0-90)	0.27	14.2	17.8	21.2

TABLE 12

Effect of Sugars on Auto-Oxidation of Ascorbic Acid Solutions at 30° C. With Oxygen Bubbled Through at Rate of 97 Ml. Per Min. at 40% Sugar or Sirup Solids

Sugar	% ascorbic acid oxidized in 2 hours
None	62.5
Low conversion corn sirup	49.6
High conversion corn sirup	
B	24.9
A	100.0
Dextrose	14.9
Levulose	32.2
Lactose	62.8
Maltose	12.6
Sucrose	24.7
Sirup, type 0	48.0
Sirup, type 0-90	49.6

Typical data are summarized in Tables 11 and 12. Of the pure sugars tested, the most efficient in retarding oxidation of ascorbic acid in solutions containing about 50 mg. percent of ascorbic acid allowed to stand quietly exposed to air at room temperature were maltose, levulose, and lactose; the least effective was dextrose. In the case of sirups, the most effective were high conversion corn sirup B and invert sirup; the least effective was high conversion corn sirup A. The sample of the latter which we tested also happened to contain the highest amount of copper. Under conditions of vigorous oxygenation, however, the order of decreasing protection was maltose, dextrose, sucrose, and lactose. The order of decreasing protection in sirups was: high conversion corn sirup B, sucrose, invert sirup, corn sirup (low conversion), and high conversion corn sirup A. In the first case oxygen solubility and rate of diffusion of oxygen were limiting factors which were not involved in the second case. These results are typical of the general trends observed in our investigation to be reported elsewhere.

Effect of Sugars on Rate of Transformation of Dehydro-Ascorbic Acid

In spite of the extensive investigations that have been reported on various aspects of Vitamin C retention in foods, we know very little about the mechanism of

ascorbic acid destruction in processed foods. As pointed out by Cameron *(1)*, until some fundamental work is applied to this problem we shall have to deal with the situation empirically. All that we know definitely is that ascorbic acid can be transformed reversibly by mild oxidation into dehydro-ascorbic acid which is still largely biologically active. Dehydro-ascorbic acid is postulated to exist either in the triketo form or in the hydrated form, but since it has not to date been obtained in pure crystalline form as a product of oxidation, our knowledge of even this reaction mechanism is not complete. The dehydro-ascorbic acid by irreversible hydrolysis of the lactone ring can be transformed into 2, 3 diketo gulonic acid, biologically inactive. The gulonic acid also has not been isolated in pure crystalline condition.

On subsequent oxidation it or dehydro-ascorbic acid is transformed into oxalic and threonic acids. Although several acceptable methods of analyis for ascorbic acid are available, satisfactory methods for the determination of dehydro-ascorbic acid and the other products, particularly as they occur in foods, are not known. Recently, however, Penney and Zilva *(15, 16)* have suggested and applied a method for the determination of gulonic acid in the oxidation products of ascorbic acid. We have applied their method to the investigation of the effect of sugars on the rate of conversion of dehydro-ascorbic acid into gulonic acid. Typical data are presented in Table 13 which shows the extent of conversion

TABLE 13

Effect of Sugars on Transformation of Dehydro-Ascorbic Acid into 2, 3 Diketo Gulonic Acid

Sugar or sirup	Concentration %	Percent Conversion	
		10 hours	20 hours
None	.....	34.5	53.0
Fructose	20.0	30.0	48.0
Dextrose	20.0	26.5	40.0
Lactose	20.0	19.0	37.5
None	.....	35.5	53.0
Dextrose	40.0	21.5	32.5
Sucrose	40.0	33.5	47.0
Maltose	40.0	28.0	37.5
None	.....	35.0	53.0
High conversion corn sirup A	40.0	32.0	43.0
High conversion corn sirup B	40.0	25.0	35.0
Glucose	40.0	29.5	40.5

of a freshly prepared 0.1% dehydro-ascorbic acid solution into gulonic acid at 28° C. and pH 5 (McIlvain's buffer).

The data presented, which were previously reported at the December 4, 1947, meeting of the Northern California Section of the Institute of Food Technologists, indicate that sugars do decrease the rate of this conversion. At a concentration of 20 percent, lactose was most effective and levulose least. At a concentration of 40 percent, dextrose and high conversion corn sirup B were most effective, and sucrose least so, initially. Later the order of decreasing effectiveness was dextrose, high conversion corn sirup B, maltose, glucose sirup solids, high conversion corn sirup A, and sucrose. It is likely that the reported losses in Vitamin C content of foods heated in absence of oxygen may be due to reactions of this type, and there is some evidence that dehydro-ascorbic acid is more thermolabile and more reactive with amino acids than ascorbic acid. More work in this field is now under way in our laboratories.

Summary and Conclusions

There is relatively little available technological information on the basis of which proper selection of sugar for use in frozen fruit products could be made other than by empirical means subject to the human error of any particular taste panel appraisal.

On the basis of such taste panel appraisal, the color, flavor, and texture retention in frozen apricots and peaches were influenced by degree of inversion of sucrose. Nectarines were less sensitive to this effect. In general sugar sirups containing more than 50 percent invert sugar are not satisfactory liquid packing media for such fruits.

Data are presented to show that sugar absorption, oxygen solubility, rate of auto-oxidation of ascorbic acid, and rate of hydrolytic transformation of dehydro-ascorbic acid, are affected by the kind and concentration of sugar used. These are but some of the basic data required to evaluate the protective effect of sugar for a particular fruit product.

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Non-Alcoholic Beverages[a]

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A review of the whole field of non-alcoholic beverages is given. A brief history, covering the origin and development of the non-alcoholic beverages is included with the description of some of the products. Precent trends in the non-alcoholic beverage industry are discussed.

The beverage industry is large, important, and is worthy of the attention of the Food Technologist. Many kinds of beverages have been devised, from time to time, in various parts of the world, yet in this country there has been so much standardization on a relatively few types that many of potential value may have been overlooked.

The American Bottlers of Carbonated Beverages is offering the Chesterman award of $1000 in cash for the most outstanding technical contribution made during each calendar year to the progress and advancement of the soft drink industry: an indication that the industry appreciates the need of research and development.

Beverages perform many services besides the obvious one of slaking thirst. Most drinks have important food value; it is hard to say, for instance, whether milk should be classed as food or beverage. Some have stimulating value and tend to keep one awake and lively because of a content of caffeine, whereas others are mildly sedative, favoring sleep. Herb teas and mineral waters sometimes have medicinal action, but today, in view of the multitude of medicines of high activity that are available, any slight medicinal function is largely ignored. In some places, however, certain drinks are still called "tonics." Perhaps the biggest argument for the use of beverages is that they constitute a treat or mild indulgence that can be enjoyed by the individual or that provides a ritual to go with sociability.

Water as a Beverage

Water is the oldest beverage and the most important. The fact that water from any source may be used for drinking purposes calls for practically every water supply being given sanitary and flavor consideration. Spring waters and deep wells may be depended upon to be clean and wholesome, but large stream or lake sources of adequate purity are becoming more and more difficult to find. As a consequence, treatment of water supplies has become standard practice nearly everywhere, often with adverse effect on the flavor. Algae growing in reservoirs in late summer, and most industrial wastes, injure flavor. As a consequence, flavor improvement of water, to make it fit and attractive to drink, has become a large and important business. The water from many city water systems, especially those along the Atlantic seaboard, is normally pleasant. In highly industrialized areas, the chlorinated organic taste often drives people to seek supplementary sources for drinking purposes and for the making of tea and coffee. Chemical and adsorbent treatments to eleminate bad tastes are being developed, however, as well as deionization and distillation, and it is expected that before long nearly every tap will yield pleasant-tasting water.

Water is generally preferred cool and many want it actually iced. No clean-cut information is available that harm comes from drinking ice water, at least when it is sipped. Physiologists point out, however, that when very warm or very cold drinks are taken with meals, they should be drunk slowly so as neither to overstimulate nor to impede digestive action. Physiologists seem not to be particularly concerned about dilution of the stomach contents by even large amounts of imbibed liquid. Generally, any advice given is in the direction of drinking more water than is usually taken.

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Carbonated Beverages

There seems little doubt that it was the English chemist Priestley who discovered that artificially carbonated water was safe to drink. He found that it had a taste similar to that of water from certain celebrated springs. The Swedish chemist Bergmann checked Priestley's findings a few years later, in 1770. It was in Philadelphia, however, whence Priestley fled, that carbonated water was first consumed in quantity, in 1785. There, a Dr. Philip Physick treated patients with carbonated water as a medicine. His pharmacist, Townsend Speakman, added fruit juices *(1)* to some of the carbonated water which then could be used as a beverage: the first "soda," "pop," or "tonic." This production of beverages in an apothecary shop led other pharmacists including Elias Durrand, also of Philadelphia, to produce a series of combinations of soda water with indigenous and exotic flavorings, and mixtures, leading to sarsaparilla, root beer, ginger ale, numerous specialty drinks and finally to the "cola" drinks. The end has not yet been reached, but the active development period seems to be over and new types have appeared less and less frequently during the past fifty years.

Up to the present, the only gas used for "charging" water appears to have been carbon dioxide.

Bottled carbonated waters are rarely drunk plain in this country, as is common in Europe, but are used chiefly as "mixers" for various drinks, both alcoholic and non-alcoholic. Some of these waters contain enough salts to produce a distinct "mineral" flavor, which "goes well" in mixtures.

It is common American practice to combine flavored, colored and acidulated sirups with carbonated water in automatic filling machines. A measured quantity of the sirup is first injected into the bottle, which is then filled with cold carbonated water. The residual air at the top may be replaced by carbon dioxide by a puff of the latter. The bottle is then capped, and mixing is accomplished by one or more inversions of the bottle. In this manner, gingerale, cola drinks and fruit flavored drinks are produced. A new item thus produced, is flavored with volatile apple concentrate, of the type invented by Howard Milleville *(2)*.

Some idea of the volume of sales of carbonated soft beverages may be gained from the quantity of sugar used, about half a million tons per year or about 3.4% of the total sugar used in this country. One manufacturer claims to supply over 100 drinks per year for every person in the land, a retail sales volume of well over half a billion dollars. There are about 6000 plants and warehouse points, virtually all of which are locally owned and operated, giving direct employment to 80,000 to 100,000 people and utilizing nearly 50,000 trucks for transportation.

The Caffein Beverages

Tea is a contender for first place as the most ancient extracted beverage *(3)*. It could have originated either in India or in China, for the shrub is indigenous to both countries. However, it has been referred to only in the Chinese literature, first about three thousand years ago. It did not reach Japan until the ninth century or Europe until the 16th.

Tea is best prepared by extraction of the leaves by very hot water. Any boiling after addition of the tea makes for bitterness and astringency, from the tannin, but strangely enough some people prefer boiled tea. Today, a large proportion of tea is sold in convenient bag form. The blend is usually adjusted for best flavor with the water available in the district in which it is distributed. The flavor of tea is much affected by the water from which it is made. Regardless of the kind of tea, whether it is the plain-dried green teas or the leaves that have been carefully fermented before drying to make black tea, it should be brewed in a pot. With pot-brewing, the water will be hotter than the critical 180° F. point, where the rolled leaves open up and good extraction ensues. The present restaurant habit of serving an uncovered cup of merely warm water for extracting the tea is doing this beverage much harm.

While tea has a pleasing though mild aroma, some producers intensify and sweeten the normal flavor by adding jasmine flowers or rose petals to cans of the dry tea. Sometimes the petals are left in the tea, but more frequently they are removed after most of their fragrance has been transferred to the tea. Even a second or third flower addition may be made to produce the highest flavor. There are localities where tea leaves are dried in an atmosphere of smoke, which imparts a mild creosoty character that some people like.

Maté or "Paraguay tea" is very popular in South America where it has been used for an indefinitely long time. The flavor is reminiscent of that of smoky Asiatic tea, but it is usually too smoky to appeal to most North Americans or Europeans. A North American beverage called "Cassina" or "Yaupon" is produced from a variety of the holly bush similar to that from which Maté is made. It has not become widely used in spite of at least one attempt at its popularization.

Coffee is another drink of considerable antiquity, going back certainly for five hundred years and likely for over a thousand years in Ethiopia and in Arabia where the wild tree is indigenous. Tradition implies that it has been used in these countries since time immemorial. The first "coffee house" appeared in England in 1650 as a sort of club or social center. It is probable that both Greeks and Turks appreciated coffee, as they still do, considerably earlier than did the English, for there is record of a coffee house in Istanbul as early as 1511.

Coffee is prepared in many ways in the different countries, and each method of preparation has its earnest proponents who maintain that their way is best. Greeks and Turks prefer strong, sweet coffee sirups, made from boiled coffee. These are sipped relatively cool, after the grounds settle. Syrians add cracked cardamom seeds to the cup of boiled coffee which is further flavored with either rose-water or orange-flower water. Americans and Scandinavians like coffee hot and of medium strength. French and Italians prefer dark strong coffee, made from heavily roasted and finely ground beans. The French, and Americans in the New Orleans area, like coffee best when it contains con-

siderable chicory, which makes for a viscous beverage of full and fruity taste. The French and some Englishmen like "Café au Lait," which is made of equal parts of strong boiled coffee and boiled milk. In some exclusive places, Spanish coffee is served, percolated with boiling milk and containing no water at all. In South American countries, strong coffee extracts are usual, made by continuous percolation. Enough extract may be made up at a time to last for a week. The extract is poured from a cruet into hot boiled milk, for cream is very scarce in those countries. This coffee, while obviously strong, is almost without aroma, causing the common expression among visitors from the North: "The South Americans know how to raise the bean but they don't know how to make a good cup of coffee."

Recently, some fairly good dry coffee extracts have been produced, which yield a beverage on mere solution in hot water, without the need of boiling, drip-brewing or percolation. These extracts have a flavor acceptable to many people, even though they are plainly low in aroma, and are so convenient that they are winning wide acceptance. In some places it is even possible to secure a cup of hot coffee from a slot machine, made from extract, with sugar and cream as desired.

The most important thing about coffee is not how it is brewed, what blend is used, or how strong it is made, but how fresh it is. No coffee is really good that has been exposed to the oxygen of the air for more than a day or two after roasting and grinding, or after the opening of a vacuum-packed container. Coffee extracts also should be relatively fresh when used.

Chocolate is another beverage of great antiquity, which originated in South or Central America. It was first tasted by Cortes when he conquered Mexico, in 1528. At that time, the richer people flavored their chocolate with vanilla and spices and sweetened it with honey or sugar. The liquid was whipped into a froth and drunk with the mouth opened very wide, the better to engulf the delightful foam. Present-day Mexican chocolate is a blend of chocolate and coffee, aromatized with vanilla. "Champurrado" is made from a thin sweetened corn meal gruel called "atole," by flavoring with chocolate, nutmeg and cinnamon.

An important use for chocolate is in soda-fountain drinks, many of which are based on chocolate sirup. Chocolate blends exceptionally well with fruit drinks on the one hand and with milk, malt and ice cream drinks on the other.

Cocoa, which is partially defatted chocolate, is widely used for both hot and cold drinks. Cocoa shells make a cheap beverage roughly similar in flavor to cocoa.

The Indians of the Amazon have another drink, comparable with cocoa, called guaraná, tasting somewhat like coffee, which has been enjoyed locally for a very long time.

Herb and Cereal Drinks

From time to time, various herb teas have been popular, sometimes of necessity, as during the American Revolution, when blackberry leaves, wintergreen (teaberry) and other substitutes were used instead of taxed English teas. While none of the herb teas, or so-called "tisanes," are actively stimulating, they have pleasing flavors. The French country people enjoy vervain tea and peppermint tea. Widely used over all of Europe is linden-flower tea (Tilia). In the Balkan countries, marjoram or sage infusions are used as stomachics, whereas Kat (catha), as also rosemary, infusions are used to combat fatigue. Pleasant tasting drinks are made from chamomile, jasmine, lavender, or dried hollyhock blossoms. Ginger tea, made from ginger and water, with or without milk, was a favorite in this country a few generations ago, as it is in India and many other countries today, often taken "for colds." Catnip tea is a favorite with children.

There are coffee imitations that may properly be classed as cereal drinks. These toasted grain preparations do not resemble coffee very closely although they are satisfactory to some people as a beverage. Decaffeinated coffee has some of the real coffee flavor, but none of the stimulation.

Thickened Beverages

Barley water, a thin gruel made from barley grain, has been used for centuries. It has been popular as a beverage among workers in the fields. The boiling of the water in its preparation has no doubt saved many lives where contaminated water was used. Somehow, a thick drink gives thirst-quenching satisfaction during work in the dust and sun that is not obtained with water alone. The food value is small but perhaps not negligible. Similar drinks are made with rice, oatmeal and flaxseed, as well as with hollyhock flowers (Althaea) and sassafras leaves (filé powder).

In the Balkans, salep, the powdered tuberous root of several varieties of orchid, is used as a thickening agent. On mixing with water and boiling, this powder swells to a thick mucilaginous mass. Salep is used extensively during the winter months, in place of coffee, among people who work outdoors. The drink, Racahout, used by the Arabs of North Africa, is a combination of salep, cocoa, sweet acorns, and potato and rice starches, sweetened and flavored with vanilla.

A delicious cold drink, made of pulverized blanched almonds, sweetened with sugar, is popular in practically every country bordering the Mediterranean. It is the special drink for young girls for such celebrations as weddings and christenings. This milk-like beverage is most pleasant, especially when flavored with anise or angelica. Where almonds are not plentiful, blanched apricot kernels may be used, or the seeds of cassaba or Persian melons, pounded in marble mortars and strained through a fine sieve.

Sirup-Based Beverages

The Koran, the sacred book of the Moslems, specifically forbids the beverage use of alcohol. The command is by no means puritanical and makes no restriction against enjoyment being obtained through beverages, provided they contain no alcohol. The result has been the development of many delightful drinks based on sirups. Most of the sirup drinks, now known the world over, originated in the Mediterranean region. The Arabs, Turks and other peoples of the Near East use many flavors that are uncommon hereabouts, which

might be surveyed with interest. Mere naming of these flavoring substances does not begin to do justice to the delightful sirups that they produce.

SYRUP FLAVORINGS OF THE MEDITERRANEAN REGION

1. Essential Oils	2. Fruits or Flowers
absinthe	apricot
anise	blackberry
bergamot	cherry
cardamom	citron
caraway	currant
cassia	jasmine
cloves	lavender
coriander	lemon
cumin	mulberry
dill	narcissus
ginger	orange
juniper	peach
laurel leaves	pear
lavender flowers	pistachio
lemon	plum
lime	pomegranate
mace	quince
mandarin (tangerine)	raspberry
mint	rose
neroli	strawberry
nutmeg	tamarind
orange	wild cherry
pennyroyal	wild orange
peppermint	
pistachio	
rose	
sassafras	
spearmint	
thyme (origanum)	

Where vanilla is needed, the alcohol-containing extract is not used, but a piece of the bean tied on a string, which may be re-used many times, until it no longer contributes useful flavor. A little licorice powder is sometimes liked. A delightful touch is often produced by the use of a flower water, obtained from flower petals by steam-distillation.

In the Balkans, sirups sometimes are diluted with plain water to make a beverage. Cooling may be done with snow, as is common in mountainous countries everywhere. Often the snow is pounded to high density and stored in caves, protected from the sun. It is transported in the summer in white wool felt bags, often an inch thick, the hard snow blocks being sawed to size. While cold beverages are often sold in stores, along with ice cream and thick, fermented milk products such as matzoon and yogurt, many are dispensed by itinerant merchants. Such a merchant may carry a large polished brass or copper jug on his back containing cold fresh spring water. This he pours from a long spout leading over his shoulder, to dilute the variety of sirups which he carries in small containers in a sort of cartridge belt.

All throuh the South American countries, street vendors prepare iced fruit drinks to order. They grind ice to the consistency of snow, mix fruit sirup with it, compress the mass, and serve it on a stick for convenient "dry drinking."

Juices

In recent years, fruit and vegetable juices have attained great popularity. These have the appeal of pleasant flavor, high food value and a notable content of vitamins. The trend is to drink for breakfast a glass of the juice of orange, grapefruit, prune, pineapple or tomato. Sauerkraut juice is a favorite in Philadelphia. Beautifully clarified apple juice is available and other juices appears from time to time. There is also a "liquefied" apple wherein the pulp is reduced to almost milky fluidity. Most fruit juices are sweet, and are otherwise attractive enough to appeal as a drink at any time through the day.

An Equadorian drink, called naranjilla, is especially delicious, recalling "ground-cherry." This is produced from a tomato-like fruit of golden color that grows on a bush. In tropical and semi-tropical countries many other delicious juices are produced, for immediate consumption, that might be widely popular if means could be found to keep them. These include the rich milky juice of the green coconut, the sweet watery juice of the ripe coconut, date palm juice and sugar-cane juice.

The "Ades"

A class of still drinks, of which lemonade is typical, has existed for a long time, both as the fresh beverage and in bottled form. The best of these drinks have a good proportion of fruit juice plus sugar. Generally, some pulp is included to make the drink cloudy. These items comprise orangeade, limeade, lemonade, as well as "smashes" and punches, all based on citrus fruits. The high acidity impresses many people with their thirst-quenching properties.

A trend has appeared from time to time to omit all fruit juice from the drink and to flavor only with essential oils, plain or terpeneless. This practice has little to recommend it, for the citrus essential oils are irritating, as well as poor-keeping, and should not appear as the principal flavor in drinks. A similar practice is the pulverizing of entire oranges, skin and all, and adding this pulp to sugared water. It is a fair-tasting drink, but far too high in oil and too low in real juice.

Drinks of Animal Origin

Fresh milk is a beverage of high food value that is of enormous popularity, especially with young people. Some think that the great height and vigor of the present generation is due largely to the widespread use of milk. It is not only taken as such, but is used with tea, coffee and cocoa beverages, and with fruit, chocolate, coffee and malt flavors in milk shakes. Combined with cream, eggs and sugar, it appears in ice cream and in the various beverages made therefrom. A childhood drink is "cambric tea," half milk and half hot water.

Fermented milks have been favorite food-beverages for ages. Some, like buttermilk and whey, have had the fat largely removed, but many of the milks soured with special cultures contain all the fat. A well-known example of fermented whole milk is "Bulgarian" or "acidophilus" buttermilk, which was popularized a generation ago, following the publication of Metch-

nikoff's enthusiastic writings. This tart beverage, while thick, is not too thick to drink directly without dilution. Some of the cultured milks, however, like yogurt, matzoon, kumiss and kefir, are so thick that they may be spooned, as a custard, or diluted somewhat with water and whipped, to make a drink called "ta'an." "Cultured buttermilk," available on the American market, is very similar to the Balkan matzoon, though perhaps a bit more tart. Some people like half molasses and half buttermilk as a drink.

In India, a favorite nightcap for children is produced from milk, heated until it develops a yellowish color and a caramel-like flavor.

Beef extracts and bouillons like Bovril, Oxo, etc., make drinks popular in the cold weather. Preparations based on malt, cocoa and milk such as Hemo, Ovaltine and Cocomalt, are food-beverages of considerable popularity. Recently, attempts have been made to push protein hydrolysates, but the strong flavors are against them. Single amino acids, such as glycine, have been suggested as ingredients of beverages.

Clam juice is a food-beverage popular in the Northeast, and a blend of clam juice and tomato juice is a favorite with many in New York City. Clam broth is now obtainable in concentrated form. For those who haven't tested it, we suggest the trial of a cup of bouillon, broth or other kind of thin soup, as a beverage, to go with sandwiches.

* * * * *

This review of the whole field of non-alcoholic beverages has been written to serve as a stimulus for broader thinking on beverage matters. We hope that it will cause initiation of items new to America and the extension or improvement of familiar drinks.

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Methods of Determining the Firmness and Setting Time of Pectin Test Jellies[a]

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The authors review pectin testing methods used in their own and other laboratories, and describe procedures now employed for estimating the setting time and jelly strength of pectins, comparing the results obtained with those secured by other methods. They present a strong case for the Ridgelimeter.

The characteristics of fruit jellies and jams which are of most importance to the consumer are the physical appearance and the taste. Many other physical qualities such as firmness, texture, elasticity, and fracture should, however, be considered if one wishes a complete evaluation of the test jellies involved with pectin standardization. There are two general methods which may be utilized in this particular problem.

The first is to provide some kind of over-all test, preferably simulating actual conditions of use, which integrates the various factors and gives the proper weight to each, but which expresses all this in one numerical quantity or rating.

The second is to follow the more classical scientific method, measuring one factor at a time, and while so doing, eliminate variables by maintaining other factors in non-critical ranges optimum for the single factor being determined.

The completely integrated or over-all test would be without question the easier course for all concerned if only suitable methods could be found which all users and producers would agree properly reflect the utility of the pectin being tested.

This becomes complicated when one considers the great diversity of products into which pectin from a given production batch may be used. Even in the field of use where jelly grade is of prime importance, the pectin may go into pure fruit jellies where the pH varies from 2.5 to 3.4, or into baker's type jellies where no fruit is used and where glucose supplants sucrose and the pH is usually below 2.5. Pectin from the same batch might possibly be sold to a confectioner who uses it in 80-83% sugar jellies with varying acidities and buffer contents. Finally, part of that same pectin shipment may go into the production of household pectin packages, which in itself is a highly complex field of use.

Thus it is evident that the "use test," for a large scale producer of pectin, is hardly in the realm of practicability, even if one considered only the preserving industries as users of pectin. In fact, the preserver who uses pectin for more than one specific product could easily get into difficulties with his own pectin stocks if they had been separately standardized for several different uses. It has seemed logical to us that uniformity in the general jelly-making characteristics of pectin was of the utmost importance to the preserver and that the least complicated methods possible should be used in insuring that desirable uniformity.

It was only after approximately twenty years of continual search that the California Fruit Growers Exchange laboratories gave up the quest for the integrated physical or use test and adopted the individual tests. Jelly firmness was selected as the most necessary and important individual factor for standardization. The

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goal was to test precisely and reproducibly the jelly firmness which a given pectin is inherently capable of producing under standardized conditions which could be easily maintained in non-critical ranges. Further, the firmness determination should require only rugged, inexpensive equipment and the test must not be time consuming.

Special test jellies need not, of course, be salable fruit jellies made under conditions identical with those of any user. Instead they have been designed to determine most accurately the one thing, jelly grade as to firmness, under controlled laboratory conditions. Should this scientific approach require further justification among technologists, consider a field other than our own where we will be less apt to have fixed ideas. Portland cement is tested for tensile strength by molding cement paste into standard briquettes, curing these in moisture-saturated air the first day and thereafter under water until they are to be broken in the testing machine. Certainly no one uses cement in just this way but all users and producers are benefited by this test which is much more precise and reproducible than would be the case if actual conditions existing in reinforced concrete construction were approximated for this tensile test of cement.

Two broad possibilities are open for testing jelly firmness. The jelly may be subjected to conditions so as to exceed the elastic limit whereby it will be permanently deformed or actually ruptured, or the tester may be such that the jelly will undergo no measurable permanent change during the test procedure. The literature on these types of testing methods is quite extensive and need not be reviewed here.

Our work has indicated that tests involving permanent deformation or rupture of the jelly are influenced by a variety of factors, each perhaps of some importance but not capable of easy control or separate evaluation. These factors are generally highly dependent upon slight variations in manipulative technic during jelly preparation and testing and their combined effects are quite apt to be confusing. Testers which operate within the elastic limit of the jelly are free from many such influences.

The trend of our work on this problem has been to refine the jelly-making method which was devised here with the assistance of H. T. Leo and was first published by Wilson *(16)* twenty years ago, then to develop the Lockwood and Hayes "Ridgelimeter" *(8)* into what is now widely known as the Cox-Higby "Exchange Ridgelimeter" *(3)*. It became necessary, along with these developments, to provide a laboratory control method for the standardization of slow and rapid setting pectins, types now commonly known by pectin users throughout the world. Since the setting time of pectin jellies may influence the grade testing methods it is desirable to consider the setting time factor before discussing the preparation and testing of sugar-water-pectin jellies.

Cole, Cox and Joseph *(2)* in 1930 showed that the rate of gelation of pectin jellies, commonly referred to as the setting time of the pectin, could have a profound influence upon the results obtained by certain laboratory testing procedures. Olsen *(10)* later published results which confirmed this contention regarding setting rate and jelly firmness. When he used a relatively slow-setting citrus pectin he found it desirable to use a large, fixed amount of acid in the test jelly, so that the pH was about 2.2. His data show that the Tarr-Baker tester readings in a 65% sugar test jelly remained practically constant from a pH below 2.0 up to about 2.7. His jellies were made by holding the pectin constant and varying the amount of acid which was added along with the pectin. If Olsen had used a very fast setting pectin he would have found a loss in jelly strength at the higher acidities, due to the fact that by his "short-boil method," the acid was present all during the cooking period and premature gelation was taking place.

A later publication by Stuewer, Beach and Olsen *(15)* illustrates how the so-called optimum pH method of making test jellies can be very misleading. Their curves for the effect of pH on jelly strength for apple pectins show a very definite optimum jelly strength near pH 3.1 with rapid decreases in strength at both higher and lower acidities. One might conclude, from those curves, that the way to get full value from the pectins would be to use them only at pH 3.1. This would also be a good argument for using a pH 3.1 buffer solution in the test jelly so as to maintain the system at the peak of its sharp optimum strength curve. Those authors realized, however, that the setting rate of the jellies, when acid was present in their test batches, became an important factor. Their curves comparing the short-boil and excess-acid methods for apple pectin jellies clearly prove that there is no optimum pH for the apple pectins when premature gelation is avoided by having the setting acid in the glass, as recommended by Wilson *(16)* six years earlier. Their jelly strength curve, when using the excess acid method, shows a jelly strength practically identical with that indicated by the peak of the optimum pH curve, and furthermore, the strength remains constant at this level from pH 1.9 up to about 2.8, where a decrease starts in both curves. The authors stated that, "Observations made here indicate that most of the difficulties involved in the routine evaluation of apple pectin jellies may be obviated by the simple expedient of preparing the jelly mixture without acid, adding an excess of acid to the glasses, and pouring the jelly mixture into these at exactly 96° C." These conclusions regarding the best method for grading apple pectins apply to the method our own laboratories have used with citrus pectins for the past quarter century.

Exchange Method for Grading Pectin

Preparation of Jellies. The method described in 1928 by Wilson *(16)* was given in more detail in 1944 by Cox and Higby *(3)* when they discussed the "Exchange Ridgelimeter" for testing pectin jellies. The actual directions for preparing the test jellies, as described in our book of standard methods, are considerably more specific than is indicated by the Cox and Higby description. A copy of the fully detailed method has been furnished to the Research Committee of the National Preservers Association and would be given here except for

space limitations. There are some amplifications to the 1944 version, however, which should be listed here.

The pan specified in the detailed method is a 4-quart stainless steel saucepan. Actually a 3.5 quart size, stainless steel or aluminum, would suffice. The pectin-sugar dispersion mixture is added to the distilled water all at once with vigorous stirring and then is allowed only a 30 second period before being placed upon the fire. The fire is so adjusted that the total heating time for the 1200 gram batch of jelly should not exceed 6 minutes. After the batch is cooked to weight and removed from the fire, the pan is tipped slightly so the liquid nearly overflows. A thermometer is put in the batch and when the temperature has dropped a degree or two, the surface of the solution is scraped free from any scum of bubbles which may form. Just as soon as the temperature reaches 95° C. the batch is poured. The first glass, a regular Hazel-Atlas No. 85 tumbler containing 2.0 ml. of preserver's acid (376 grams tartaric acid per liter of solution) has previously been placed in the setting time apparatus as illustrated in Figure 1. The remaining

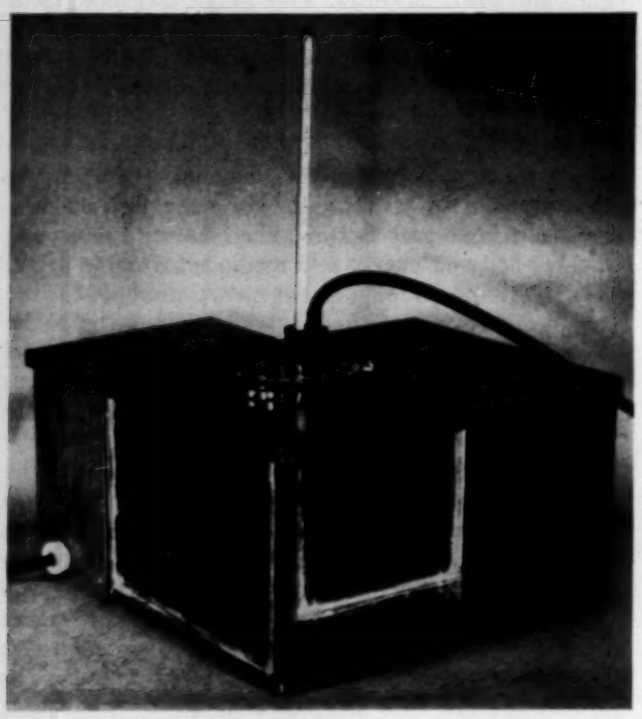

FIG. 1. Water bath apparatus used for determining setting-time of pectin test-jellies at 30° C.

three glasses are the calibrated ones furnished with the instrument and which have been fitted with paper "sideboards" as mentioned in the Cox-Higby *(3)* article. The sideboards are made by using ¾ inch gummed paper tape and allowing ½ inch to project above the glass. These latter three glasses should contain 2.6 ml. of the standard acid solution since their volume has been increased by the "sideboards."

A glass rod in the tumblers will permit stirring as the batch is poured but, as Olsen *(11)* has pointed out, stirring in the glasses is not necessary if one pours very rapidly. The glasses should be filled to the top of the "sideboards."

Determination of Setting Time for Pectin Jellies. Although the setting time of pectins has been mentioned in pectin literature for about two decades, we have seen no precise method given for its determination. Olsen, Stuewer, Fehlberg and Beach *(12)* in 1939 described what they termed a quantitative method. We believe the method which our laboratories have used since 1937 permits more precision and should be described in this connection.

The apparatus used is illustrated in Figure 1. It is a water bath having glass windows on opposite sides and a support for a jelly glass placed between the windows so that when the tank is filled to the overflow outlet with distilled water at 30° C., the jelly glass is surrounded with water almost to the top and is held in place by a clip provided for the purpose. The light placed behind the rear window enables one to observe the jelly through the front window. Circulation of the water is obtained by a slow stream of air bubbling beyond the field of vision between the two windows. Temperature adjustment of the bath for subsequent batches may be made easily be adding water of the proper temperature. The 30° C. level chosen is such that radiation cooling is almost off-set by heating due to pouring the hot jelly.

The jelly batch is poured as has been described above. A stopwatch is started just as soon as the first glass is filled and then the remaining glasses for Ridgelimeter testing are poured rapidly. Close attention must then be given to the first glass which is in the 30° C. water bath.

The glass in the water bath is given a slow, easy twist at intervals, and observed through the window in the front of the tank. The set will be seen to progress from the bottom to the top of the jelly. At first, the particles of jelly will be seen to rotate only in the direction in which the glass was turned. As the jelly starts to set, it will be seen that the jelly particles in the lower part of the glass will rotate first in the direction in which the glass was turned, and then will move back in the opposite direction. The watch is stopped when the last portion of the jelly at the top of the glass can be seen to move back in a direction opposite to that in which the glass was turned. The interval between the starting and stopping of the watch is the setting time of the jelly, usually referred to as the setting time of the pectin.

The so-called setting times of most commercial pectins, in the 30° C. bath and at pH values between 2.2 and 2.4, will be less than 5 minutes. The relation between setting times at 30° C. in the water bath and in air at 20-25° room temperature, is shown by Table 1.

The setting times shown in Table 1 pertain, of course, to the specific test jellies involved and to their pH and sugar concentration. It is obvious, as Leo, Taylor and Lindsey *(7)* have stated, that as the pH of the test jellies is raised, these setting times would become longer. However, contrary to the contention of those workers, pectins do have inherent properties affecting the rate of set of their jellies when measured at fixed conditions of temperature, sugar concentration, and acidity *(4)*. For instance, when using the standardized jelly making technic just described, Exchange slow-setting pectins

TABLE 1

Relation Between Times of Set for Exchange Standard Test Jellies, in Air at Room Temperature and in a Water Bath at 30° C.

Setting Time in Minutes	
Air at 20-25° C.	Water Bath at 30° C.
1.00	0.90
2.00	1.65
3.00	2.25
4.00	2.80
5.00	3.25
7.00	4.00
10.00	5.10
14.00	6.30

produce jellies whose setting times, by the present method, fall within the band of 3.25 and 4.00 minutes, while under the same conditions rapid-setting pectins set as rapidly as 0.10 minutes. The preserving industry is well acquainted with the characteristic difference between the behavior of these two types of pectin when used in juices and at higher pH levels.

Determination of Jelly Firmness with the Exchange Ridgelimeter. The Ridgelimeter used for measuring the firmness of test jellies is shown in Figure 2. The details

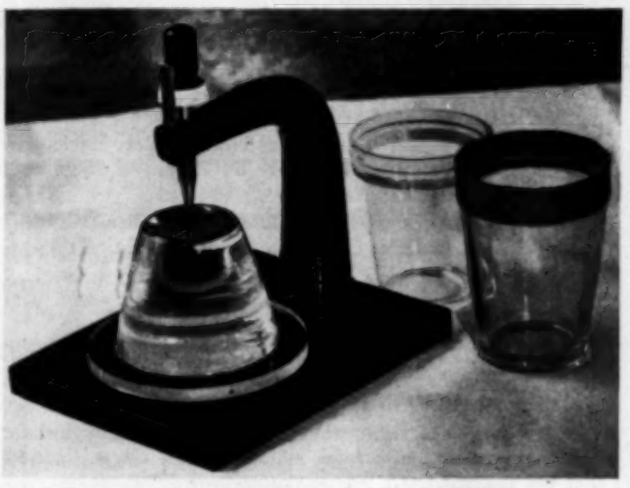

FIG. 2. The Exchange Ridgelimeter, showing a jelly in the measuring position. Calibrated glasses are shown with and without "sideboards."

of operation for this instrument have been published by Cox and Higby *(3)* and additional information about the preparation of jellies for the standardized test have been given in this present paper. Our original publication on the Ridgelimeter did not emphasize the importance of checking the scale of the instrument occasionally, especially if new glass plates were substituted for those originally furnished.

An excellent checking gauge can be made by cutting a ¾ inch stainless steel rod to exactly 2.50 inches in length. When this rod stands on the glass plate and the point of the micrometer screw just touches its top, a scale reading of exactly 20.0 should be obtained. If the reading is not 20.0 the vernier and scale can be adjusted so as to produce this reading by using the set screws provided on the instrument. It has been found that the best results are obtained when the tip of the micrometer screw is about 1.0 mm. in diameter.

The calibration curve which we use for the instrument in grading regular pectins (both slow and rapid setting) is reproduced in Figure 3. This curve shows that we consider a sag of 23.5% to be normal, as was mentioned by Cox and Higby *(3)* in 1944.

The curve of Figure 3 does not apply to the grading of low methoxyl pectins. This of course is to be expected since the rheological properties of the two types of gels

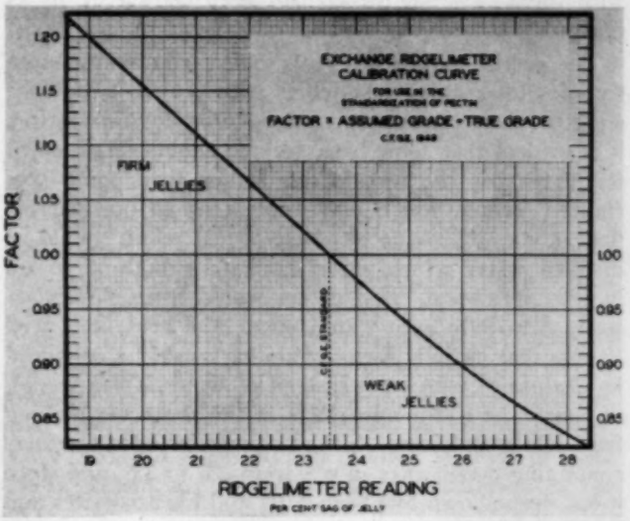

FIG. 3. Calibration curve for regular citrus pectins graded by the Exchange Ridgelimeter.

are different, as has been shown by Cheftel and Mocquard *(1)*. The curve used in our laboratories for the standardization of low methoxyl pectin is nearly parallel to the one of Figure 3 and crosses the line of factor 1.00 for normal firmness, at 20.5% sag. The gel batch for testing low methoxyl pectin is entirely different from that used with regular pectins, and is described elsewhere *(5)*.

Discussion

The subject of the present symposium, "Pectin Standardization," implies the comparing of pectins with some standard, or actually it means the insurance or guarantee of uniformity. The actual choice of a "standard" for pectin jellies has been considered by some as a difficult problem. We believe that uniformity in jelly strength is of the greatest interest to the preserver or jelly maker and that the Exchange Ridgelimeter with its easy and precise measure of jelly firmness permits the standardization of pectin to a uniform jelly grade.

The standard of firmness we selected for calibration purposes, a sag of 23.5% for the particular test jelly described here, was based upon twenty years experience with hand grading of jellies which the preserving industry considered normal. Owens and Maclay *(13)* in 1946 stated that:

> "The value of 0.235 for the unit compression used as the standard of firmness by Cox and Higby yields a value of about 3 g./cm.^{2} for the shear modulus. It might be well for the industry to consider some such standard value with a standard procedure, in order to establish objective standards by which pectin may be sold. From the work presented here, a more reliable measurement would result if a slightly stiffer gel were chosen for the standard. A value of 4 g./cm.^{2} would be more desirable."

However, a year later Owens, Porter and Maclay *(14)*, while discussing the grading of pectins and the use of certain instruments for that purpose, stated that "A shear modulus of 3 g./cm.^{2} was chosen as a standard, and the results suggest that it be considered by the pectin industry as a basis for grading their commercial products." This value for the shear modulus coincides with the 23.5% sag which had been chosen by us as representing a standard jelly.

The pectin literature reveals many arbitrarily chosen technics for making test jellies with pectin. However, since the main concern now seems to be standardization, it is evident that some basic technic should be accepted for preparing the test jellies, rather than some specialized technic which may apply only to one narrow field of use. The aqueous phase of test jellies should be distilled water which would contribute nothing to aid in jelly formation. If a pectin would form a satisfactorily firm jelly with water, sugar and acid, it is well known that its jelly forming ability would be enhanced by the use of fruit juice instead of water. This would, of course, be to the advantage of the preserver making fruit jellies and jams. Since commercial jelly products containing pectin vary in pH from 2.0 to 3.5, and since it has been shown by Olsen *(10)* and his associates and by hundreds of our own tests, that there is a region of constant jelly strength for water jellies at pH 2.2-2.4, there seems no reason for wishing to get out of that pH region in making test jellies. Lampitt and Money *(6)* in 1937, using a tester with a vane inside the jelly, found that the jelly strength was practically constant from pH 2.0 to about 3.0. They studied testers which broke or tore the jelly and reported that "None of these methods appears to be measuring the 'true' strength of the gel." Test jellies at higher pH regions, even in the presence of "artificial juices," are subject to influences which should be avoided in choosing a basic recipe for use in insuring uniformity or standardizing. Twenty-five years ago Macara *(9)* showed that pectin jellies at high pH, particularly with fruit juices, were very slow to attain maximum firmness. He pointed out that high acid was useful in hastening development of maximum firmness. This factor is of great importance to pectin producers where dozens of pectins must be graded daily and where factory production must not be delayed by unnecessary jelly testing complications.

Finally, it is our opinion that the test jelly should be made with water, should have sugar at 65% concentration, and with acid in the glasses to bring the final pH down to 2.2-2.4. The instrument for grading should be rugged, inexpensive, simple in design, easily and quickly operated. It should operate within the elastic limit of the jelly. The Exchange Ridgelimeter seems to meet these qualifications although in common with most others, this instrument does not lend itself to measuring the sag in jellies previously "set" in other than the standard containers. Owens and Maclay *(13)* in comparing the Ridgelimeter with the Tarr-Baker tester type and with their own rather complicated "Rigidometer," show that the Tarr-Baker tester gave results on duplicate gels varying as much as ± 5% while the other two instruments kept these variations within ± 2%. Owens, Porter and Maclay *(14)* later reported that the Tarr-Baker instrument "was more time consuming and less reproducible" than their Rigidometer and the Exchange Ridgelimeter. They renamed this latter instrument "Sagometer" to avoid confusion with the name of their own instrument which they had subsequently devised. Referring to the Ridgelimeter, therefore, they say, "The Sagometer which measures percentage sag of a gel is rapid and precise, and can be calibrated against the rigidometer to give values for the shear moduli."

More than fifty Exchange Ridgelimeters are now in use, outside our organization, in this country and abroad. Although this fact leads us to believe that the instrument and method as here described meet practical requirements, we are still alert to any possible improvements.

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Grading Pectins[a, b]

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A comparison is presented between the use of acid during the preparation of test jellies for grading, and the use of salt mixtures for this purpose. Synthetic juice buffer salt solutions are described as preferable since they more nearly resemble the actual products.

A study *(1)* has just been completed which indicates that more emphasis is needed upon the preparation of test jellies for grading purposes than upon the method of testing. Two types of measurement, breaking strength and sag, were made in this study of grading and two methods of adding acid, in the glass and before boiling, were employed. Buffer salt solutions were also used in one phase of the study. Certain of these phases of study appear to have produced results which warrant presentation in a symposium on "grading."

This paper will consider two questions: (one) "Should acid be present during the preparation of test jellies for grading?" and (two) "Should salt mixtures be used in jellies for this purpose?" The paper will present a comparison of the results of the two types of measurement upon jellies made by the two methods of adding acid over a pH range 3.5 to 2.5, or 2, as the case may be, in the presence and absence of buffer salts.

Should Acid Be Present During Cooking Period?

This question has been considered by others in the past *(2, 5, 7)*. For instance, the point has been made by Stuewer et al. *(7)* that the "excess-acid" method of grading, where an excess of acid is placed in the glass, is more simple because it eliminates the complication of first determining the optimum pH of gelation. Actually this pH is of negligible importance because pectin manufacturers have standardized production methods which allow them to predict quite closely the optimum conditions of acidity for gelation. If necessary, they could illustrate the trend of curves for elasticity or breaking strength of jellies, cooked with and without acid, from the various types of pectins they produce with a fair degree of accuracy. These curves would appear to be of value to the trade in deciding which pectin to choose for a certain job, or for continued assurance in the uniformity of the product. A few tests would soon indicate to the pectin producer whether his product was showing variations from normal as to optimum pH for gelation, sag, etc. This would obviate the necessity for determining the effect of small increments of acid over a wide range of acidity in the case of each and every pectin. In this, study, however, the interest is in what happens to the physical characteristics of sag and breaking strength when the acid is present or absent during cooking.

When the effect of the manner of introducing the acid upon jelly strength and sag was studied upon jellies containing 0.67 percent commercial apple pectin, results were obtained as in Figure 1. Point-to-point curves are drawn to better indicate variations in breaking strength. The solid line indicates that a smooth, typical jelly-strength curve will be obtained when various amounts of acid are present during the cooking period. Again, a uniform curve (long dashes) is shown for sag measurements upon the jellies cooked with increasing amounts of acid. On the other hand, more erratic results were obtained from both strength (short dashes) and sag measurements (dot-dash) upon jellies produced by pouring, with rapid stirring, hot sugar-pectin mixtures upon varying amounts of acid placed in the jelly glasses. While the trend is toward gels of lower strength as more acid is present, the strength under the conditions of the "excess-acid," which adjusts the pH of the jelly to 2.07, would not appear to be satisfactory for grading purposes. Sag at pH 2.07 of the jelly made by placing the acid in the glass compares favorably with the sag at pH 3.1 of the jelly made by a 4.5-minute cooking with acid (normal boil procedure), however, this is thought to be largely a matter of chance.

Contrary to the results shown by Figure 1, for a rapid-set apple pectin, results obtained upon jellies containing 0.50 percent of a slow-set citrus pectin shown in Figure 2 would seem to indicate that jellies for grading by means of strength measurements might be made either by cooking with acid (solid line) or by pouring the hot sugar-pectin mixture into acid in the glass (short dashes). The strength value used for grading could be either that at the optimum pH (2.85) or that made with "excess-acid," at about pH 2.1, without being too far out-of-line, provided the proper factor be used for aligning the two sets of results. The slow-set citrus pectin differs from the rapid-set apple pectin in the degree of coagulation or pregelation which takes place during about 4.5 minutes of cooking with acid. No pregelation occurs when cooking gels containing the slow-set citrus pectin; the slight decrease in strength as more acid is added is due to hydrolysis. Sag measurements upon the jellies where the acid has been added to the glass show uniform values below pH 2.75 (dot-dash) and these could be readily adapted for grading this type of pectin, but less uniform values (long dashes) were obtained where the acid was present during cooking.

Figure 3 illustrates that when acid is present while cooking the jellies containing 0.57 percent of a citrus rapid-set pectin, the breaking strength reaches a maximum at pH 3.15 and decreases gradually, but uniformly, as more acid is added (solid line). When the acid is present in the glass and the other ingredients, which have been cooked, poured thereon, fairly uniform values

[a] This paper consists largely of data and discussion taken from a more extensive study on grading of pectins which will be published as Bulletin 272 of the Delaware Agricultural Experiment Station.

[b] Published as Miscellaneous Paper No. 52 with the approval of the director of the Delaware Agricultural Experiment Station. Contribution of the Department of Chemistry, May 7, 1948.

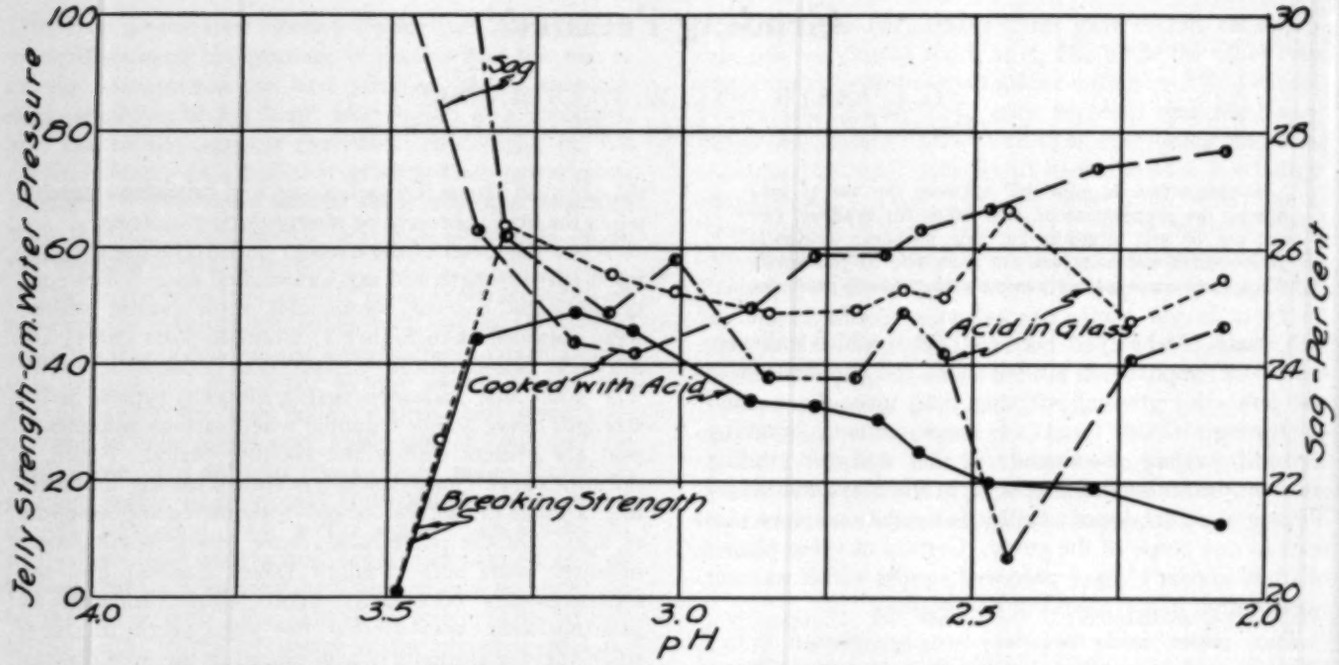

FIG. 1. Effect of adding acid before and after cooking on jelly breaking strength and sag—apple pectin.

are obtained for breaking strength as higher concentrations of acid are present until the point is reached where coagulation tendencies are paramount (short dashes). When the acid is added in the glass, the tendency is for the strength to be much greater at the lower pH values, than the strength in the normal pH range of gelation (3.0 to 3.3) in commercial usage. Also, the strengths are much greater than by the "normal-boil" procedure (solid line). If sag measurements are made upon jellies containing this same citrus pectin more uniform results are obtained when the acid is present during cooking. The broken line in Figure 3 shows that while a fairly regular curve is obtained, any grade determination at pH 2.1 based on the sag measurement would be out of line with a grade determination at pH 3.

In summarizing, it is indicated that grading could be based on both jelly strength and sag values in the case of the apple pectin when acid was present during cooking, but not when acid was placed in the glass and the rest of the jelly mixture poured into it. In the case of the slow-set citrus pectin it would appear that the jelly strength measurement may be used in grading with either method of making the jelly and, while there might be some question on the use of sag measurements in the

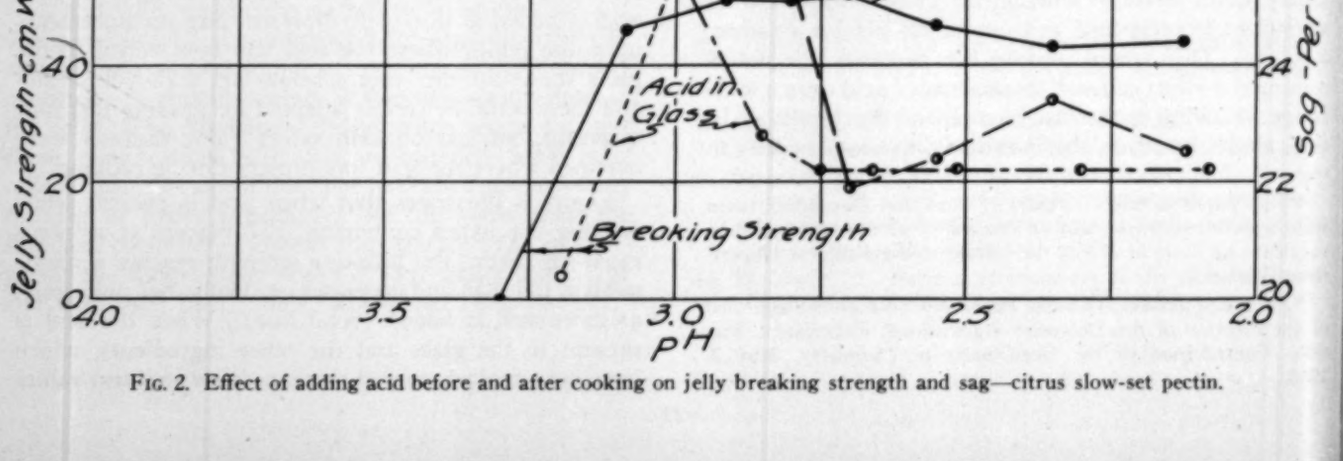

FIG. 2. Effect of adding acid before and after cooking on jelly breaking strength and sag—citrus slow-set pectin.

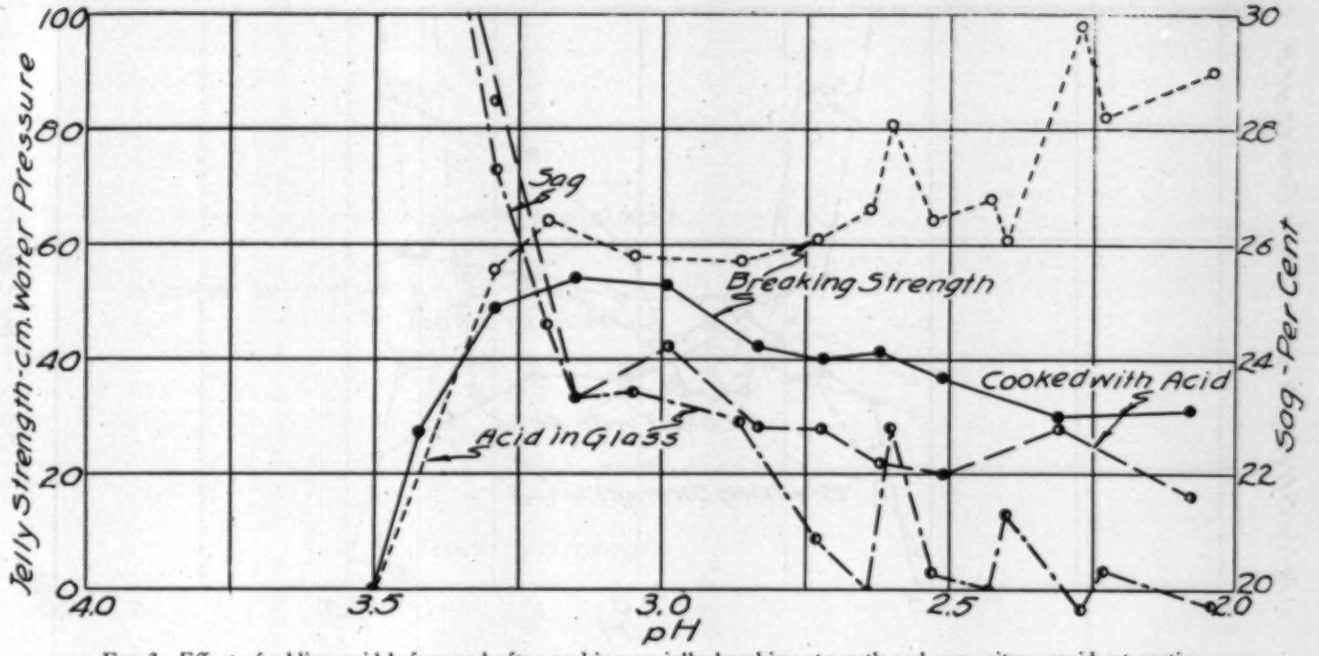

FIG. 3. Effect of adding acid before and after cooking on jelly breaking strength and sag—citrus rapid-set pectin.

case of the jellies cooked with acid, apparently sag measurements can also be used. However, in the case of the fast-set citrus pectin, the results would indicate that breaking strength alone should be used in measurements for grading pectins of this type. Only the jelly strength measurements upon jellies made where acid was present during cooking may be considered satisfactory as indices for grading purposes in the case of all three pectins. The results indicate that both the method of measurement and the method of making the sample gels influence results. Obviously, something must be done differently in order to obtain a better correlation of results.

Should Salt Mixtures Be Present During Grading?

Olsen et al. *(6)* claim the so-called optimum pH can be modified through use of a synthetic juice, a mixture of buffer salts and acids which represents the average analysis of a series of strawberry juices. The composition of their mixture follows:

Potassium Phosphate	1.535g
Potassium Citrate	6.33
Sodium Citrate	0.985
Calcium Citrate	1.19
Magnesium Acetate	2.02
Citric Acid, anhydrous	25.00
Sucrose	40.00
Water to make one liter.	

Despite the conclusion of Olsen et al. *(6)* that the preparation of jellies containing these salts and 65 percent soluble solids at pH 3.1 is completely satisfactory, it was decided to determine the effect of variation of pH over the range 3.5 to 2.5 using the same three pectin samples discussed in the last section.

The results of preliminary study were so promising that it was decided to make a more detailed examination of the effect of a salt mixture, similar to that of Olsen et al. *(6)* with the exception that less citric acid (17g.) was used in order to adjust the pH to 3.5. Following this, there were added increments of acid (0 to 5ml. 50 percent tartaric) to provide a series of jellies in the pH range 3.5 to 2.5. (Magnesium tartrate was used in place of magnesium acetate through error).

The pectin was dispersed in two liters of the salt solution as a 1.0 percent solution in the case of the apple pectin; as a 0.75 percent solution in the case of the slow-set citrus pectin; and as a 0.85 percent solution in the case of the rapid-set citrus pectin. This was done by mixing the necessary pectin in each case with 100g. sucrose and then dispersing the mixture in the synthetic juice and diluting to two liters. This made a sugar-salt-acid-pectin mixture containing about 12 percent soluble solids. Two series of jellies were made for each pectin. The pH of one series was adjusted to vary between 3.5 and 2.5 before cooking and each jelly was boiled to 150g. final weight; in the other series, each jelly was boiled to 145g. (about 67.2 percent soluble solids) in order that the final weight would also be 150g. when the cooked sugar-pectin juice concentrate was poured on the 5 ml. of acid solution of varying strength in the glass. The strength of the acid solution was varied according to the desired pH. Each jelly was made as follows: Sucrose, in an amount sufficient to raise the total solids added to the jelly mixture to 97.5g., was placed in a one and one-half quart sauce pan; acid was added; water was added sufficient to adjust the original weight to a uniform value; then the jelly mixture was boiled to 150g., or 145g., as the case may have been; and, finally, the jelly mixture was poured into standard 5-5/32 ounce, Hazel-Atlas No. 2750 tumbler-shaped jars. In the series of gels where 5.0 ml. of acid solution was present in the glasses, the other ingredients, properly concentrated, were poured into the acid and the whole stirred with a glass rod for 15 seconds. After skimming,

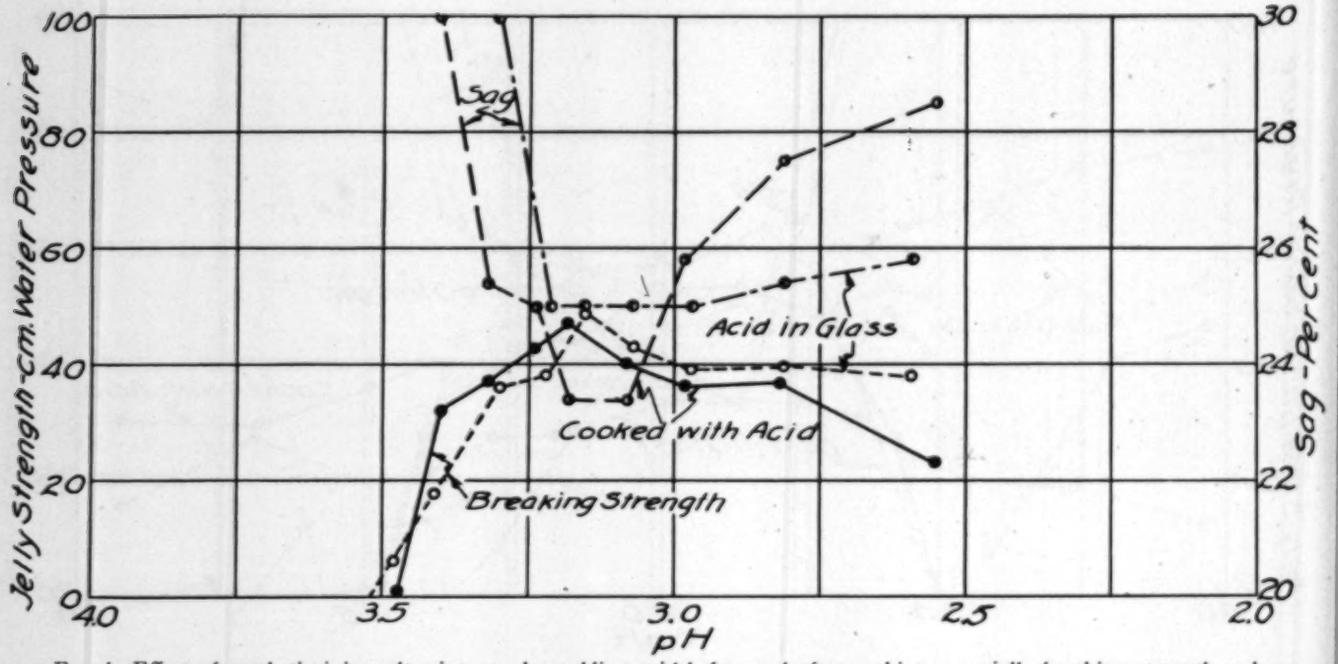

FIG. 4. Effect of synthetic juice salt mixture when adding acid before and after cooking upon jelly breaking strength and sag—apple pectin.

the jellies were held partially immersed in a water bath at 26° C. for 20 hours. The jelly strength was measured while the jelly remained in the glass, the jelly was then turned from the glass and the sag measured with the Exchange Ridgelimeter but not according to Cox and Higby's *(3)* directions because of insufficient jelly volume. In the modified method, a standard block was used under the glass plate in order to raise the jelly sufficiently to measure with the adjustable screw of the Ridgelimeter. Sag was calculated in accordance with the method of Lockwood and Hayes *(4)*; the original height of the jelly in the glass was measured, the height of the jelly after two minutes from the time it was turned out of the glass was easily measured from the screw setting on the Ridgelimeter, and the percentage of sag was calculated from the difference. The jelly was returned to the glass and the pH measured using a glass electrode. Jelly breaking strength and sag values appear plotted against pH by the point-to-point method in Figures 4, 5, and 6 for jellies made from the three pectins in the same order as they were previously tested.

The curves in Figure 4, indicating the data taken upon jellies made with the apple pectin, differ from those in Figure 1. Where the acid was present during

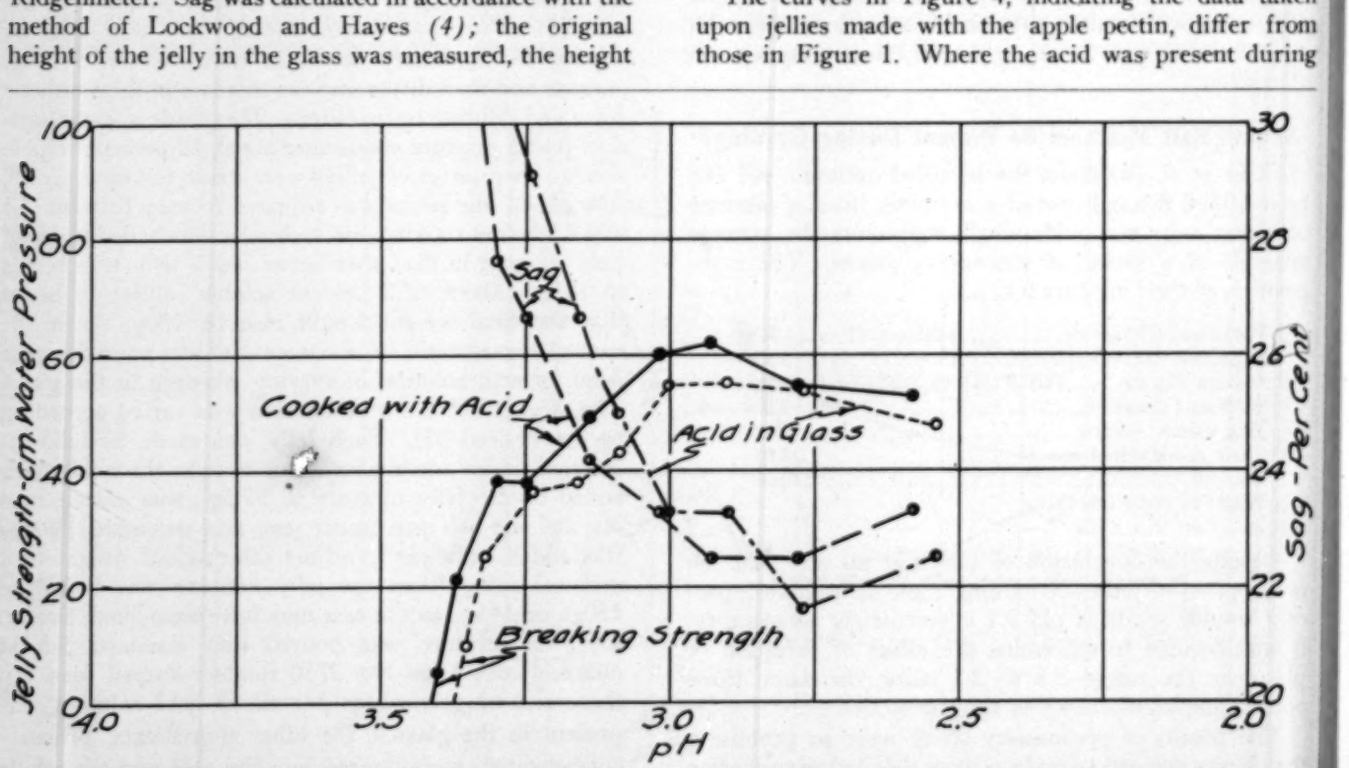

FIG. 5. Effect of synthetic juice salt mixture when adding acid before and after cooking upon jelly breaking strength and sag—citrus slow-set pectin.

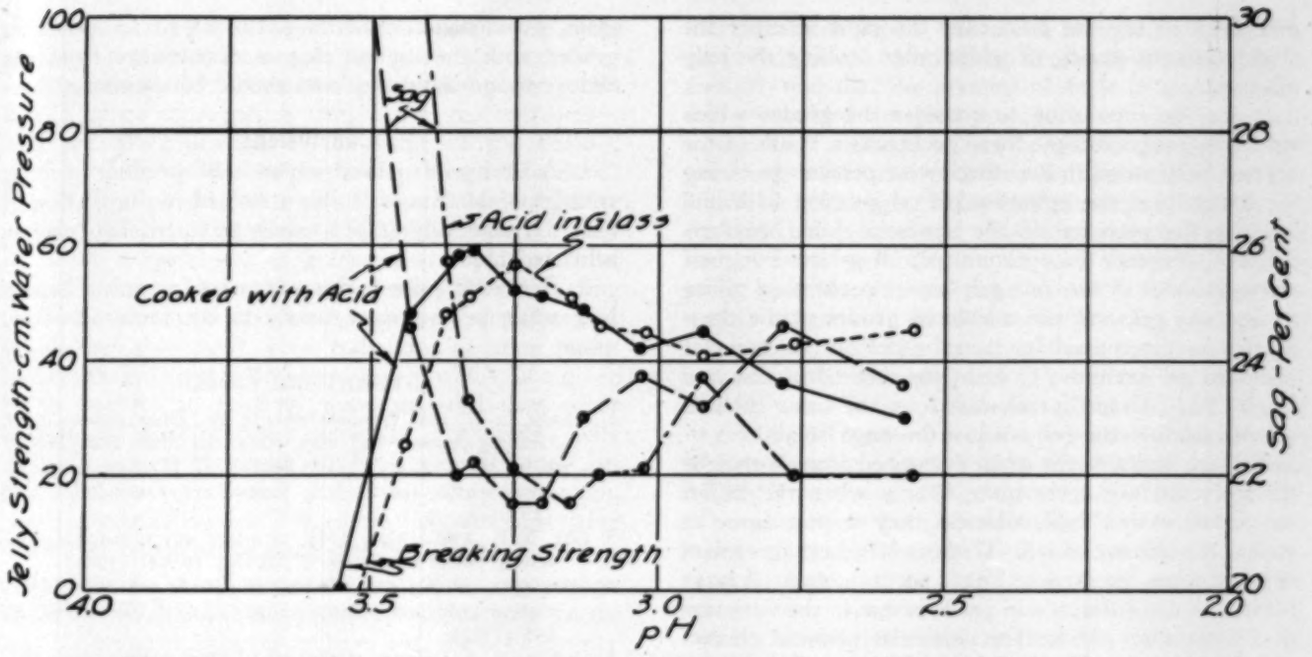

FIG. 6. Effect of synthetic juice salt mixture when adding acid before and after cooking upon jelly breaking strength and sag—citrus rapid-set pectin.

the cooking of the jelly, the lines follow the same general trend in both figures. There is an optimum pH of gelation and then the strength (solid line) decreases gradually. Sag (long dashes) increases with increased acidity somewhat more rapidly in the presence of the high concentration of salt. Contrary to the values indicated in Figure 1, the data illustrated in Figure 4 for jellies made by pouring the hot, concentrated jelly mixture upon the acid in the glass show closer agreement with the values for both strength and sag obtained on jellies made with acid present during cooking. When the acid is in the glass, a similar type of curve is obtained for both strength (short dashes) and sag (dot-dash), after the optimum strength is reached the values decline gradually as the acidity is increased.

The strength values for jellies made with the slow-set citrus pectin are also very nearly the same when the synthetic juice was used (Figure 5) whether the acid was present during cooking (solid line) or added later by having it in the glass (short dashes), the former being found slightly stronger. On the contrary, in the absence of buffer salts, it will be recalled that higher strengths were obtained if the acid was added in the glass. The curves for both sets of the sag measurements are somewhat more uniform when salts are present, but show much less sag at higher pH values, Figure 5, than in their absence, Figure 2. In other words, the initial pH of gelation is raised by having the high concentration of salt present.

The value of the presence of the buffer salts is shown again when the jellies are made from rapid-set citrus pectin. The curves in Figure 6 show a slight increase in the initial and the optimum pH when both strength and sag measurements are considered. The greatest strength

TABLE 1

Effect of Synthetic Strawberry Juice (Buffer Salt Solution, pH 3.5) upon Grading Values

Sample	Conc. of Pectin in Jelly, %	Method of Adding Acid	No Buffer Salt				Buffer Salt Solution, pH 3.5			
			Optimum pH	Jelly Strength, cm. H_2O Pressure	Sag %	Grade (Based on Strongest Gel)	Optimum pH	Jelly Strength, cm. H_2O Pressure	Sag %	Grade (Based on Strongest Gel)
Apple Pectin	0.67	Present during cooking	3.18 3.08	49	.... 24.2	96 93	3.18 3.18	47	.... 23.4	95 99
		In glass	2.44 2.44	67	.... 20.7	116 109	3.15 3.22	49	.... 25.0	96 92
Citrus Pectin (Slow-set)	0.50	Present during cooking	2.70 2.70	52	.... 21.9	133 139	2.93 2.93	62	.... 22.5	146 136
		In glass	2.85 2.75	64	.... 22.2	148 138	3.00 2.77	55	.... 21.6	137 140
Citrus Pectin (Rapid-set)	0.57	Present during cooking	3.15 2.08	54	.... 21.6	119 123	3.32 3.25	59	.... 21.5	125 125
		In glass	2.27 2.27	98	.... 19.5	164 135	3.25 3.15	57	.... 21.5	123 125

and the least sag are practically the same whether the acid is present during or added after cooking the jelly mixture.

It may be interesting to consider the grades which would be assigned these three pectins as a result of the highest jelly strength and the lowest percentage of sag for the jelly at the optimum pH of gelation with and without the presence of the synthetic juice mixture. Table 1, presents the optimum pH of gelation, highest strength value of any one gel, lowest percentage of sag of any one gel, and the calculated grades of the three pectins as determined by using established factors for standard gel strength *(1)* or by the method of Cox and Higby *(3)*. Grade is calculated on the basis of total soluble solids in the gel, not just the sugar solids.

Table 1 indicates the grades obtained from both jelly strength and sag agree quite closely when the buffer salt solution was used, whereas they do not agree as well in the absence of salt. Without salt, best agreement is found when the slow-set citrus pectin is used. A large portion of the difference in grade is due to the variation of the optimum pH for the particular physical characteristic being measured; these differences in pH are negligible where the synthetic juice mixture is present. Adding the acid before boiling produces the most regular curves whether the salt is present or not. For this reason and because the procedure is more comparable with major factory usage, it would seem to be the one which should be used by the pectin manufacturer for grading. Adding "excess-acid" after boiling, or in the glass, sufficient to adjust the pH to 2.0 to 2.3, produces grades with the highest degree of variance from the factory usage standpoint and should be abandoned.

Conclusions

1. A series of jellies which will produce a jelly strength—pH curve is to be preferred to duplication at one pH, especially at pH values produced by "excess-acid" technique.

2. Synthetic juice buffer salt solutions are of benefit in grading pectins more nearly to the terms of actual usage.

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The Preservers' Stake in Uniform Pectin Grading[a]

E. EVERETT MESCHTER[b] and LOUIS J. LATAILLADE[c]

The need for uniform grading of pectins is discussed. A method for preparing a standard jelly and physical tests for its evaluation are given. A detailed method suggested by The National Preservers' Association Research and Technical Committee represents an objective approach to the problem of pectin grading.

There are many references in the literature that contain data and present methods and procedures by which pectins can be graded. And yet the preservers, who use more than 70% of the commercial pectin production in this country, have not been heard from. I would like to present the preservers' stake in the controversial issue of pectin grading. Let me, first of all, explain how I happen to speak for the preserve industry on this problem, rather than leave it up to the pectin industry where, perhaps, it rightly belongs.

Early in 1946 the National Preservers Association appointed a Research and Technical Committee whose purpose is to investigate and attempt to solve problems of the industry which are of interest and concern to all members. One such problem, for example—the fading of the rich, red color of strawberry preserves upon room temperature storage—is being investigated under a cooperative research project directed by Dr. Kertesz at the New York State Agricultural Experiment Station at Geneva, N. Y. Another problem under investigation is the fate of DDT spray residues in grapes and grape products. There is no doubt that an attempt to establish a uniform method for grading pectins is within the scope and purpose of this committee and is a problem of importance to every purchaser of commercial pectin. Therefore, we would like to illustrate the need for the establishment of a uniform pectin grading procedure from the preservers' point of view and, suggest a concrete starting point for constructive discussion toward that end.

During the war shortage of pectin the American Preserve Co. was forced to purchase brands and types of pectin which it had never heard of before—and never hopes to again. These pectins were all labelled 100 grade, 150 grade, or some other nondescript value which had meaning only to their manufacturers, but they were so different in their physical characteristics and sugar supporting capacity that it was impossible to substitute one for another in a commercial preserve or jelly batch without considerable adjustment of the formula.

[a] Presented before the Eighth Annual I. F. T. Convention, Philadelphia, Pa., June 8, 1948.

[b] American Preserve Company, Philadelphia, Pa.
[c] Tea Garden Products Company, San Francisco, Calif.

On one particular occasion it was found necessary to purchase pectin from an apple processor who had never made pectin before. The actual production of the pectin was of minor consequence compared to the controversy over the determination of the value of the finished product. How would one go about making a "standard" jelly? How would you know it was "standard" once you've made it? There was no legal or scientific basis on which a settlement of grade value could be made. Only the sense of fairness on the part of the contracting parties was able to bring such a difficult situation to a satisfactory settlement. Even today this situation exists. Preserves find certain brands of pectin which, in a given batch, require 10 to 20% more material than other brands of similar stated grade to give equal jelly strength values under identical plant conditions. Instances have been found where a commercial pectin would grade as low as 72% of the jelly strength value of another pectin of the same specified grade.

It certainly seems reasonable to expect any article of commerce as important to another industry as pectin is to the preserve industry, to have a standard of identity by which its value can be graded by the purchaser on the same basis as it was graded by the seller.

The problem of non-uniformity of various brands was presented to the Research and Technical Committee for consideration, and it was decided to determine how wide-spread the difficulty was. A questionnaire was prepared and sent to all members of the Preservers Association. Of the 22 members who had sufficient technical data to return answers, one-third said they used more pectin in their formulas than they did 7 years ago, but apparently this was only the case when using certain brands. It seems that few members evaluate commercial pectins for themselves, most everyone relying on the grade stated by the manufacturers. Members indicated that more information was desired on the pectin which they purchase, particularly as to setting time and grade over a range of acidity common for fruit jelly. The demand for some sort of uniform method of testing and evaluating the pectin was unanimous, and the Committee was thus encouraged to carry the investigation further.

Letters were then written to the pectin manufacturers and the problem explained to them requesting that they make available their standard jelly making technique and method of evaluating the test jellies. It is interesting to note that no two pectin manufacturers used the same method of making a test jelly. No two manufacturers used the same technique on the evaluation of the test jelly, and no two manufacturers used the same method of calculating grade from the observed tests. It is little wonder from this that the preserver finds difficulty in substituting one pectin for another in his formulas.

The Committee has contacted many of the pectin manufacturers and find that they too are anxious to find a standard method for pectin grading. Information on this subject has been collected from many sources and if any of these sources hear their own material being returned to them, then it is freely admitted we are guilty of plagiarism. It is strongly felt that the literature and papers presented here today contain a great deal of valuable information toward uniformity of method, and that the greatest obstacle in uniting them into a standard, accepted procedure is prejudice against competitors' methods or inertia against change of one's own long established practice. So, the method that we would like to suggest today is an attempt to summarize consensus of opinion and consolidate the good points of many of the methods now in use, always bearing in mind, however, that any point chosen is in close agreement with commercial jelly manufacturing practice. It is suggested with the idea that it is a simple, workable and accurate technique, and is presented at this time primarily as a basis for discusion. It is designed for use in grading all types of pectin which are suitable for use in the manufacturing of 65% fruit jellies as defined by the Department of Agriculture for which standards of identity have been established. It does not necessarily apply to the so-called low methoxyl pectins capable of gelation in absence of sugar, nor to pectins which have specific uses in confectionary, leather tanning or pharmaceutical applications.

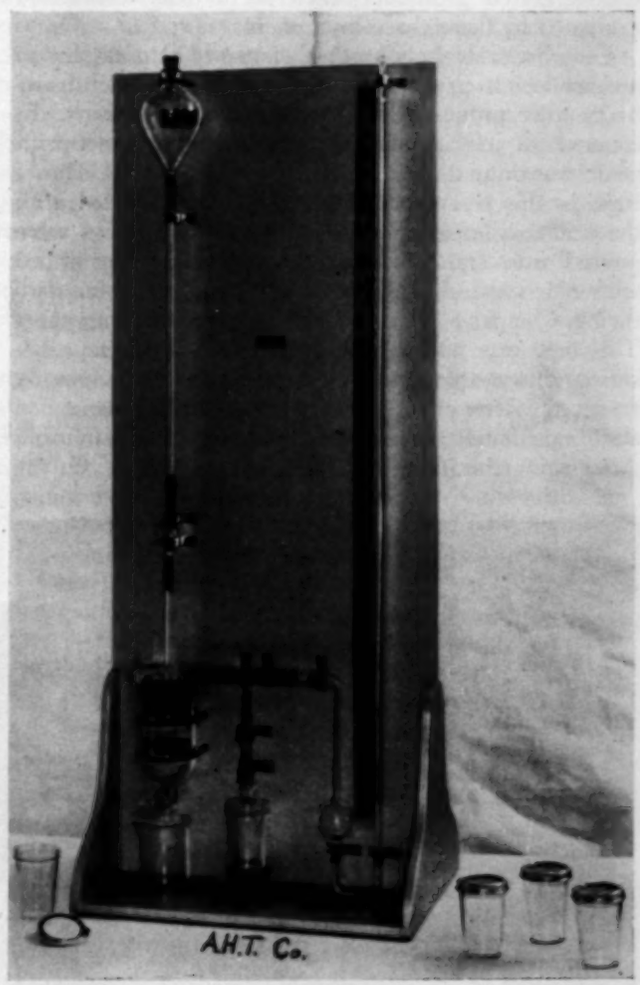

FIG. 1. Tarr-Baker Delaware Jelly Strength Tester. The jelly strength, which is indicated on the water manometer (at right), is determined by measuring the force (air pressure) required to break through the jelly surface under standardized conditions, using a plunger with a flat surface face of controlled diameter (Luer syringe lower center). Air pressure is created by displacement of air in chamber (at lower left) by liquid from reservoir (upper left). (Courtesy Arthur H. Thomas Co.)

This procedure was outlined at a meeting of the Research and Technical Committee several months ago for the purpose of evaluating the present instruments available for pectin jelly testing, and to determine if any one instrument favored any particular type or brand of pectin. The instruments chosen for study were the Tarr-Baker tester, developed at the University of Delaware (Fig. 1); the Ridgelimeter, developed at the California Fruit Growers Exchange (Fig. 2); and the Rigidometer, a development of the Western Regional Research Laboratory (Fig. 3).

The investigation was carried on independently by the authors at the Tea Garden Products Company, San Francisco, California, and at the American Preserve Company, Philadelphia, Pennsylvania. From time to time, the authors collaborated on their findings.

For this study several pectin manufacturers were requested to supply a sample of their standard commercial pectin, which, by their production control methods, was accurately graded. The nine pectins provided, included two slow set citrus pectins, five rapid set citrus pectins and two apple pectins. Then, using the suggested procedure, each pectin was tested and evaluated by these three instruments.

Each pectin was thoroughly dispersed into a solution and made into a jelly in a medium of synthetic strawberry juice proposed by Olsen and his co-workers. In commercial jelly production, pectin is in contact with an acid medium during the entire cooking period. Therefore, in this test procedure, the jelly was boiled with the acid to simulate plant conditions. The jellies were poured into standard ridgelimeter glasses, equipped with sideboards of Minnesota Mining and Manufacturing Company "Wet or Dry" brand masking tape. This tape was preferred over gummed paper tape because it was more easily applied and more easily removed. After three minutes each jelly was skimmed and immediately covered with 2 ccs of paraffin oil. After aging the jellies for 18 to 24 hours at 30° C., the top strengths were measured on the Tarr-Baker tester. The excess jelly was then cut from the top of the glass with a cheese cutter, and modulus in tension was measured on the Rigidometer. Each jelly was then removed from the glass and the modulus in compression was measured on the Ridgelimeter. A third glass of jelly which was not cut by the Rigidometer vane was sliced into seven mm slices for slice strength evaluation on the Tarr-Baker tester. Thus, two measurements of rigidity and two measurements of rupture strength were made on each jelly batch.

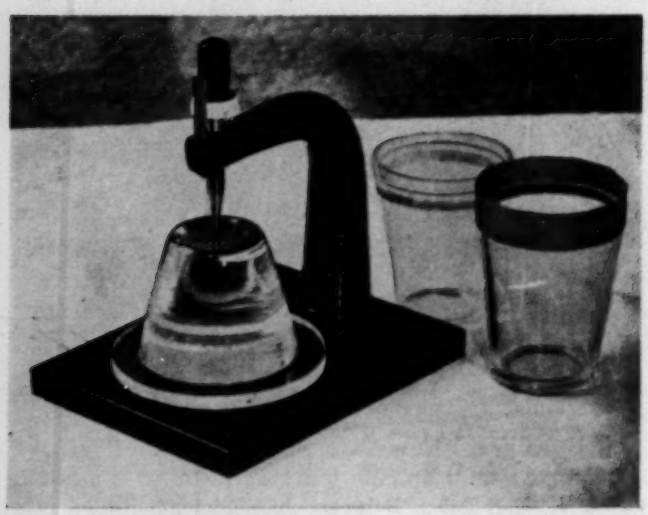

FIG. 2. California Fruit Growers Exchange "Ridgelimeter." Jelly is poured into glass equipped with paper collar or "sideboard" above rim of glass (at right). When jelly is set, "sideboard" is removed and excess jelly cut off with cheese cutter. Jelly is inverted onto glass plate and amount of sag is measured on scale when instrument pointer touches jelly surface.

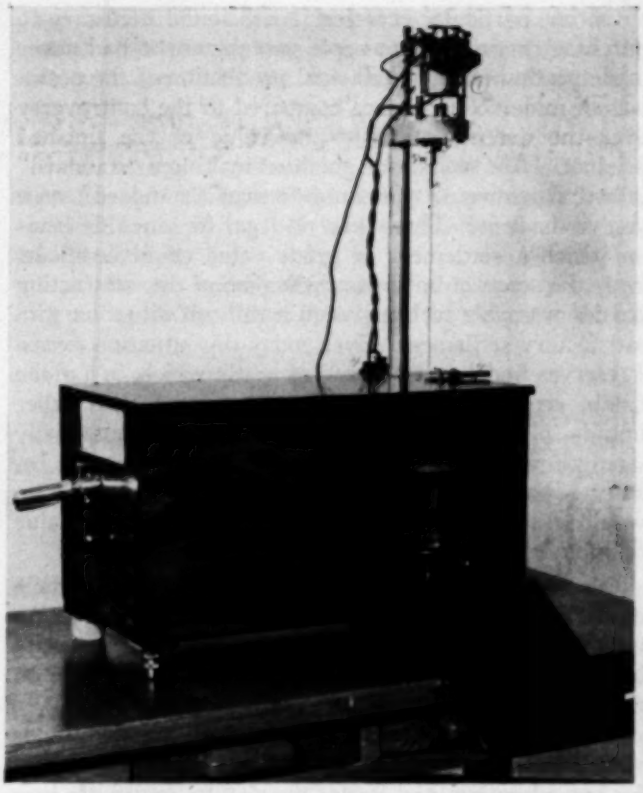

FIG. 3. Western Regional Research Laboratory "Rigidometer." Shear modulus is measured by inserting vane on bottom of wire into jelly (in box) and applying twist of 10° in torsion wire with synchronous motor (upper right). Deflection of vane in jelly is indicated on chart scale (at left end of box) by the reflection of light beam (from light source left center end of box) by mirror attached to upper part of vane.

(Courtesy Western Regional Research Laboratory)

Results of this investigation seemed to indicate the following:

1. There seemed to be a variation of as much as 30% from the highest to the lowest grade of the commercial pectin samples submitted whether evaluated by a strength measurement or a rigidity measurement.

2. There was a close correlation between grades determined by the Exchange Ridgelimeter and the Western Regional Laboratory Rigidometer. They seem to measure identical characteristics of jelly. This is in agreement with the findings of others.

3. There seemed to be a wide variation of grade over the pH range of fruit jelly of 2.8 to 3.4 when grade was determined by either strength or rigidity measurement. This difference in grade over the pH range was greater in some types of pectin than in others.

4. When some pectins were graded over a pH range the rigidity increased with decreasing pH, but the increased rigidity was not necessarily reflected in increased breaking strength and in some instances rigidity increased with decreasing breaking strength, particularly at pH values of 3.2 and below.

5. There appeared to be greater reproducibility of measurements of top strengths of jellies when paraffin oil had been added to the surface, compared to jellies allowed to age covered by a lid. This may be due to the prevention of condensate returning from the lid to the jelly surface.

6. The greatest reproducibility of strength measurement, however, was obtained by the testing of jelly slices.

7. Apparently the rate of pressure increase in the Tarr-Baker tester is extremely important for strength measurements. Variations in grading with this instrument amounted to as high as 15% when rate of pressure increase was changed from 1 cm. of water per second to 3 cms. of water per second.

The original purpose of the investigation was to determine if one particular method of grading favors one type of pectin over another. Apparently it does. Any method of grading based on rigidity appeared to favor most citrus rapid-set pectins, or pectins which tend to be brittle in their character. A rigidity evaluation tends to lower the grade of most apple pectins because of their greater elasticity. However, increased rigidity is not necessarily reflected in increased breaking strength. Therefore, it does not seem possible to assign a fair grade to all pectins on the basis of a rigidity measurement alone. The inference is not that pectins graded by a rigidity measurement are not fairly graded. Preservers of this country have been too well satisfied with the quality and uniformity of their results with these pectins to criticize this method for production control. But the average preserver does feel that a pectin graded by a rigidity method has been satisfactory because at a particular standard percentage of sag the breaking strength has also been satisfactory, and not primarily because the attributes of jelly of standard sag-value contributes greatly to desirable qualities of that jelly.

One may well say, "let's grade apple pectins on breaking strengths and citrus pectins on rigidity." Unfortunately, mixtures of several types of orange, lemon or grapefruit pectins yield various characteristics of sag in relation to their breaking strength. And there can be no sharp line of demarkation between a so-called elastic jelly and a rigid jelly.

Preservers are primarily interested in breaking strengths. This characteristic determines whether or not a jelly will retain its mold on pouring from a container, whether or not it will stand abuse in shipping and, most important, how the jelly will react on a piece of buttered bread when spread with a knife. A rigidity measurement gives valuable descriptive information, but jelly quality can be considered satisfactory over a wide range of rigidity. Jelly quality can not be considered satisfactory over such a wide range of breaking strength. Perhaps the *relation* of rigidity to breaking strength may be an important criterion by which jelly character can be described. If pectin were graded on rigidity alone, then a preserver could conceivably receive pectin which yields a brittle agar type of jelly which would be entirely unsatisfactory for commercial fruit jelly. If graded on breaking strength alone, the preserver could conceivably be supplied with pectin which yields jelly of satisfactory breaking strength and yet be so elastic as not to form a shimmering mold of the container when poured out for table use.

It would, therefore, seem necessary to grade all pectin on the measurement of *both* breaking strength and rigidity. Pectins are all different. We must stop talking about pectin and start talking about pectins. The steel industry has specifications for tensile strength, rigidity, elasticity and malleability. Each steel has advantages for a particular purpose. This is also true of pectins. We can not expect one measurement to completely describe pectin characteristics any more than we can expect tensile strength alone to tell an engineer the adaptability of a given steel for a bridge span or a structural support. Likewise, values of breaking strength and rigidity of pectin jellies describe different characteristics of these jellies, and increase our knowledge of the adaptability of a pectin for a particular use.

Preservers are interested in the evaluation of pectin jelly quality in their every-day production. It would seem reasonable then that any instruments used for grading evaluation should also be capable of telling results of pectin usage under commercial production conditions. This is the only way that a preserver could tell if commercial production reflects the value of the stated grade.

For the measurement of breaking strength, our investigation has shown that greatest reproducibility is obtained by the use of jelly slices. This technique has the decided advantage to preservers that the area of the container from which the sample was taken is of no significance. Consequently, uniform results can be obtained on jelly slices from 6 oz. glasses or 30 lb. pails (Fig. 4). The principle of the Tarr-Baker tester can

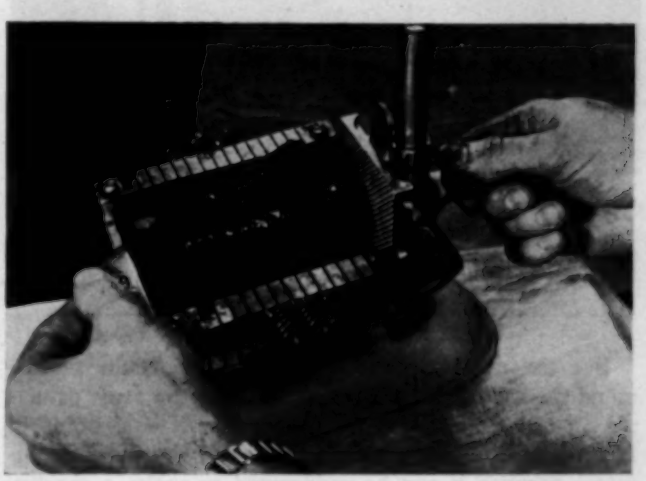

FIG. 4. Jelly slicer designed to cut slices of uniform thickness from portions of jelly from any size container. An adaptation of a butter cutter, the blades are .006 in. machinist feeler gauge stock under spring tension 7 mm. apart.

probably be developed to measure breaking strength with satisfactory accuracy. Particular attention must be paid to the rate of pressure increase, maintenance of clean plunger and barrel surfaces, and evenness of slices used for measurement. Perhaps many of these difficulties could be overcome with the development of a mechanical instrument similar to the one developed during this investigation. This mechanism is presented for consideration since we believe it may eliminate much of the difficulty in breaking strength measurements. It is not new, but merely a modification of Stoloff's device used for surface strengths. Stoloff placed the jelly sample on a dietary scale and read from the dial the pressure necesary for a plunger to break through the jelly surface. To this device we have added a constant speed motor driving the standard Tarr-Baker plunger head. The test jelly is placed on the pan of a tare beam balance. The motor drive is supported over the jelly and the plunger driven into the sample at a constant speed. The pressure increase on the jelly is reflected directly on the deflection of the scale pointer. Break-through is sharply indicated by a drop in the scale pointer. The rate of pressure increase is constant. Friction is not a factor since the balance pan is supported on knife edges. Results can be reported directly in grams per square centimeter of plunger area. The mechanism can be constructed for less than $100 in any machine shop from simple specifications and needs no arbitrary standardization since it is purely mechanical and measures directly in units of force (Figs. 5 and 6). For convenience we have called it the "Stolmotor" device.

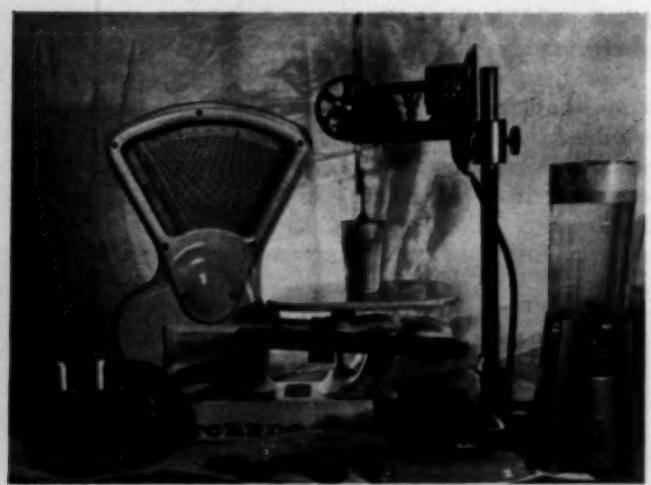

FIG. 5. Equipment suggested for jelly strength evaluation. From left to right: 4 quart Stainless steel saucepan with stirrer, tare beam balance, "Stolmotor" device, jelly slicer, and Waring Blendor.

For the measurement of rigidity we would recommend the use of an instrument based on the same principle as the Rigidometer. From a preservers' point of view this principle has an advantage over the Ridgelimeter because it can be redesigned to make a rigidity measurement on jelly in any package of any shape. It has already been shown to compare favorably in accuracy and characteristic of measurement with the Ridgelimeter, and would seem to afford a convenient means of determining rigidity.

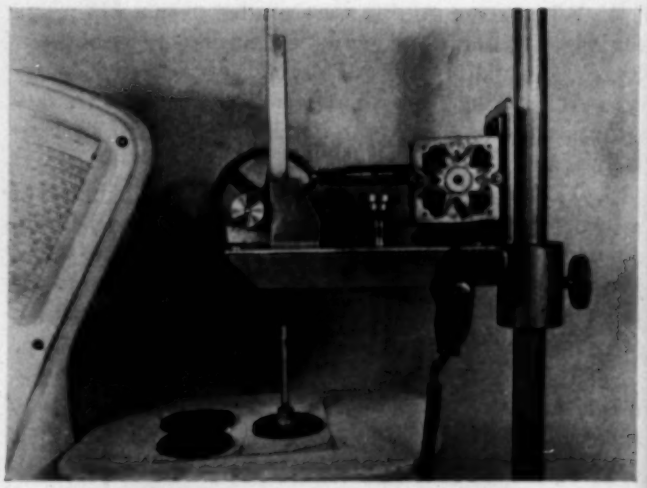

FIG. 6. "Stolmotor" device developed during the investigation. Essentially a constant speed motor with speed reduction to drive standard Tarr-Baker Tester plunger head into jelly at rate of 11 mm. per minute. Breaking strength of jelly is indicated by a sharp drop in scale pointer (left). Highest weight reached is reported as breaking strength.

It is not likely that any definite decisions can be made at a meeting of this size. We appreciate the cooperation of the pectin manufacturers in attending this meeting so that all phases of the problem can be openly discussed. It is impossible to evaluate information and ideas of other workers which will be presented at the meeting today without an opportunity of trying them out for yourselves. We, therefore, suggest that this afternoon a committee be formed to investigate and formulate a uniform jelly-making method, a uniform jelly-testing procedure, and a uniform grade-calculating formula by which all pectins suitable for 65% fruit jelly shall be graded.

We suggest that this Committee be composed of several representatives from pectin manufacturers and several representatives from the National Preservers Association. In such a group all factions interested in this problem would be represented and we feel confident that a method can be worked out by which all pectins can be fairly and accurately graded.

. . . .

METHOD FOR GRADING PECTIN

(Suggested by National Preservers Association Research and Technical Committee)

Equipment:

1. Waring Blendor with 1 liter glass cup and stainless steel blades, equipped with rheostat. This equipment has a universal motor. (The speed is proportional to the voltage.)
2. Four-quart copper bottomed stainless steel sauce pan with 1 side handle. (We found Thermic Ray made by Norris in San Francisco to be very good. Revere does not make a 4-quart with a single side handle.)
3. 50% Citric Acid Solution
   Dissolve 500 grams anhydrous U. S. P. citric acid in distilled water in a 1 liter volumetric flask. Make up to mark. This solution contains 50 grams anhydrous citric acid in 100 cc. solution.

4. Olsen Salt Solution—modified to contain 47.3 grams citric acid instead of 62.3 grams. The final pH of this solution is then 3.4 and attains the correct pH of the first jelly without further adjustment. pH of subsequent jellies can be adjusted by addition of further acid solution.

FORMULA FOR CONCENTRATED SYNTHETIC STRAWBERRY JUICE
(Olsen's Salt Solution)

Ingredient	Formula	Amount 1 kg.
		grams
Distilled Water	H_2O	800.0
Sodium Salicylate	$NaC_7H_5O_3$	0.32
Salicylic Acid	$C_7H_6O_3$	0.21
Potassium Phosphate	K_2PO_4	3.83
Potassium Citrate	$K_3(C_6H_5O_7) \cdot H_2O$	15.7
Sodium Citrate	$Na_3(C_6H_5O_7) \cdot 2H_2O$	2.03
Citric Acid, Anhydrous	$H_3(C_6H_5O_7)$	47.3
Magnesium Acetate	$Mg(C_2H_3O_2) \cdot XH_2O$	5.07
Calcium Citrate	$Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$	3.80
Distilled Water	H_2O	q. s.
	Total Weight	1000.00

Add ingredients in the order given, or if already weighed out for use, add as a mixture. Make up to 1000 grams with distilled water and stir mechanically until dissolved. Use 134 grams per batch of jelly of 1000 grams. Keep juice in a well corked bottle since exposure to air has some effect in causing precipitation of salts.

The Salicylic Acid and Sodium Salicylate are added to prevent mold.

5. Gas burner or electric hotplate—adjusted to boil off water from 1000 gram batch at rate of 100 grams between 2 and 3 minutes.
6. Stirring rod—tared with pan.
7. Hazel Atlas tumblers No. 85 (used as standard Ridgelimeter glass by California Fruit Growers Exchange) equipped with "Sideboards" of Minnesota Mining and Manufacturing Company 1½" "Wet or Dry" Masking Tape.
8. Spatula with sharpened edge to cut 7 mm. slices of jelly.

Procedure:

1. Determine approximate grade:
   Make a 1% solution of pectin and determine its viscosity. Calculate approximate grade by formula

   $\text{Log } y = ax + .2$ where

   y = relative viscosity at 26° C.

   x = grade or sugar supporting capacity of a unit weight of pectin

   a = variable factor (.6 for citrus pectins, .7 for apple pectins)

2. Prepare a pectin solution containing 3250 jelly units in 1,000 grams

   For 5 grade liquid pectin: Dilute 650 grams of concentrate to 1000 grams with distilled water.

   For powdered pectin: Weigh out an amount of pectin determined by dividing 3250 by the assumed grade of the pectin. (For 150 grade use 21.66 grams, for 100 grade use 32.50 grams.)

3. Make certain of complete solution of pectin.

   A. Place approximately 800 grams of distilled water @ 50° to 60° C. in tared Waring Blendor cup. Adjust rheostat to low motor speed and cautiously add the 3250 jelly units of dry pectin to the mixing cup. Allow to run for two minutes, gradually increasing speed of Blendor as viscosity of solution increases.

   B. After running three minutes or until pectin particles are no longer visible, return the Blendor cup to the scale and add distilled water to a final solution weight of 1000 grams. Return the cup to the Blendor and mix thoroughly at a speed as great as possible without loss over sides of cup.

   C. This solution is such that 650 jelly units are contained in 200 grams of solution.

4. Preparation of Jelly:

   A. Prepare 4 jelly batches—at pH 3.4, 3.2, 3.0 and 2.8.

   B. Weigh out 650 grams of sugar for each batch, subtracting from the sugar weight the grams of other solids to be added to the batch.

      1. Use the apparent refractive sugar solids of the salt solution as 7 grams solids.
      2. Each cc. of acid solution contains ½ gram anhydrous acid.
      3. Pectin contained in the 200 grams of solution equals the weight of weighed pectin sample divided by 5.

   C. Place the tared stainless saucepan on the balance. Weigh out 200 grams of pectin solution. Add 134 grams of Olsen's modified salt solution. (Omit salt solution when testing liquid apple pectin concentrates). Add sufficient acid solution to adjust the pH to the desired value (jelly pH 3.4 equals 0 acid; 3.2 equals 4 cc. acid; 3.0 equals 10 cc. acid; 2.8 equals 20 cc. acid). Add sufficient water to bring the total weight to 1100 grams minus weight of sugar to be added.

   D. Place pan with liquid ingredients on burner adjusted as in paragraph 5 under "Equipment." Stir occasionally to prevent burning. When boiling commences, add the sugar. Stir constantly until all sugar is dissolved. Boil until weight is reduced to 1000 grams. Pour into glasses described in paragraph 7 under "Equipment." Allow to stand for 3 minutes and skim. Immediately add 2 cc. paraffin oil to surface.

5. Measurement:

   A. Age jellies for 18 to 20 hours @ 30° C.

   B. Remove sideboards and slice off excess jelly with wire cheese cutter. Measure shear modulus on two glasses with instrument similar in principle to Rigidometer. Measure breaking strength on 7 mm. slices cut from third glass on instrument similar in principle to Tarr-Baker tester.

   C. A standard jelly has a shear modulus of 3 grams per sq. cm., and a slice breaking strength of 60.7 grams per sq. cm. of plunger area.

The Need for Standardization of Pectin[a]

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REVIEW PAPER

Some of the physical and chemical variations of pectin are described. The need for practical tests and some of the instruments for making them are discussed.

Nature has imposed a wide variation in the raw materials used by the food manufacturer. This makes the standardization of these products difficult, yet it is necessary to standardize in order to assure a uniform final product.

We have seen commercial pectin improve from the cloudy, dark solution of the first manufacturer, Douglas, containing a greater percentage of impurities than of pectin itself, to the present highly refined product meeting National Formulary standards. At the same time the use of pectin has emerged from an adulterant to conceal the inferiority of less fruit ingredient to a recognized ingredient necessary to obtain a satisfactory quality in jellies.

Perhaps the first significant effort toward development of standards for pectin for food use came with the heavy purchases for Lend-Lease purposes at the beginning of World War II. The quantity involved was approximately equal to production at the time. There was much speculation as to what it was actually being used for. One idea was that it was being nitrated to form an explosive for use in detonating block-busters. Another theory (this one facetious) was that it was to be used to jell the surface of the English Channel so that the invasion forces could march across. The quantity appeared quite sufficient! Seriously, England had been forced to reduce the fruit content of preserves and needed the pectin to make up the deficiency in the wartime substitute. Also, at the then current rate of sinkings, in order to guarantee delivery, about twice the quantity had to be shipped.

In obtaining samples and quotations on the pectin which various manufacturers would supply, the wide variations both in grade and purity, became apparent. Since British preservers would be receiving products of various manufacturers without direct contact with the source, it was necessary to develop a standard test for grade.

Early in 1942 tentative specifications were prepared, and submitted to various interested parties for comment. Reviewers in general stated the proposed specifications were "all right, BUT . . . ," the coments were more lengthy than the specifications!

Now we can possibly agree, here among ourselves, that pectin should be standardized, and then spend the rest of the afternoon arguing on the method to be used, only to conclude that because of this lack of a method acceptable to our various ideas we should not attempt to set a standard.

Standardization leads to stabilization and too often this leads to stagnation. Pectin technology is a growing field of science and I would not want to see it stagnated, but I believe we have arrived at the point to consider whether or not pectin should be standardized for jelly manufacture.

A few years ago a representative of a pectin manufacturer made the statement that pectin should not be standardized as variation in grade was the only means of competing since the price was already pretty well standardized. This manufacturer would not have been willing to accept scrip instead of U. S. Treasury notes in payment for pectin as a means of the preserver meeting competition. If we can accept the principle of standardization then we are a step toward the details.

Now pectin varies in many ways. It is not a definite chemical entity, but any preparation represents a mixture of more or less related compounds. Of first consideration is molecular size. Any representation of this property is a statistical average. The true molecular weights of the individual molecules present in a given preparation may spread over a wide or over a narrow range. It may have a large proportion near the average or an even distribution throughout. In referring to the molecular weight of pectin it is necessary to keep in mind how it was determined and whether the numerical average or the weight average was determined by the particular method of measurement employed.

Similarly, any representation of the degree of esterification is an average value. Distribution in the degree of esterification can also vary from molecules resembling pectic acid to highly methylated molecules. There is another type of distribution possible with esterification and that is within the molecule itself. The ester groups may be scattered on alternate carboxyls or grouped at one end of the molecule with a marked effect on chemical properties. In addition, there are impurities and perhaps added ingredients. However, it is not so much these specific differences with which we are concerned in standardizing pectin as it is their outward manifestations on the properties of gel strength, setting time, and solubility.

Much diversity of opinion exists in regard to method of measurement of gel strength, first as to the method of preparation of the test gel and then, the method of testing the strength of the gel. Most directions read as if they were taken from a cook book. Certainly the use of laboratory equipment instead of pans and spoons, gets away from the kitchen methods used in the past.

The question of the pH value at which test jellies shall be made requires discussion. First let it be known that the grade as stated does not necessarily mean that a preserver is going to use it at that ratio to sugar in his product. He may derive more or less jellying power from the fruit used and the pectin is added to correct the deficiency. Furthermore, he may want a tender jelly for local consumption and a tougher jelly for distant shipment. The pH of the jelly will vary with dif-

[a] Presented before the Eighth Annual I. F. T. Convention, Philadelphia, Pa., June 8, 1948.

ferent fruits and this will affect the strength of jellies obtained with the same amount of pectin and with different pectin preparations.

If we are to standardize pectin we must agree on a pH for test purposes realizing it may not be used at this pH, and that the pH at which it is used may affect the strength of the jelly. The pH should be close to that of most common jellies. If we have a pectin standardized at a certain pH then we can better correct for the factor of a different pH in the jelly. Opposition to use of a stated pH in jellies in favor of the optimun pH might just as well follow an analogous line of reasoning and conclude that pectin cannot be standardized, because the fruit juices in which they are used contain a varying amount of pectin, rendering such standardization impossible. Let the preserver know what he is buying and he will be in better position to make suitable corrections in formulation. Whether or not a stated concentration of salts should be used in test jellies is debatable.

The method of testing the strength of the test jelly will receive much discussion. There are old-timers with an educated little finger who will want to continue to rely on it. The finger test is fairly reliable with an experienced person when comparison is made with standard jellies. The inexperienced do not trust it. Moreover, it does not give an accurate estimate of the degree of variation from a standard jelly.

The Tarr-Baker or Delaware jelly tester has been used perhaps more than any other instrument for testing jelly strength. The criticism has been made that the apparatus has not been standardized and that one does not get readings in C. G. S. units. It is a relatively simple process to calibrate a given Delaware tester from the ratio of the diameter of the head of the plunger to the diameter of the plunger itself, taking into consideration the weight of the plunger. A calibration chart can be made using standard pectin. By reduction to C. G. S. units, a standard pectin would not need to be used but it would be necessary to agree to the pressure representing 100 grade pectin under standard conditions of making the test jelly. Using standard 100 grade pectin as agreed upon by suppliers under Lend-Lease we found the instrument in use at Eastern Regional Research Laboratory to give us a reading equivalent to 93 g. per square cm. for a standard jelly.

The Bloom jelometer has been used for some time by the gelatin and glue industry. It is a more standardized instrument than the Delaware testes, being constructed to rather rigid specifications rather than assembled from available parts. This equipment has not heretofore been available to preservers because of limitation on its sale to the gelatin industry only, but it is now offered to preservers as well. It has not been generally studied by pectin people except those who are also interested in gelatin. It is worthy of consideration along with other jelly testing equipment.

Another instrument which has been suggested is the California Fruit Growers Exchange "Ridgelimeter." This equipment measures the per cent sag in a jelly and will therefore give a reading less favorable to a jelly with a high elastic modulus than one with a low elastic modulus.

The British have made use of the B. A. R. tester. B. A. R. stands for the British Association of Research for Cocoa, Sugar Confectionery and Jam Trades, London. This measures the torque on a blade immersed in the jelly produced by a weight and is another means of measuring elastic modulus.

Recently, Owens, Porter and McClay *(1)* of the Western Regional Research Laboratory have developed a device along the lines of the B. A. R. tester which measures shear modulus. Cheftel and Mocquard *(2)* have also designed an apparatus along similar principles which measures elastic modulus. Another instrument has been developed in Kodak Research Laboratories by Goldberg and Sandvik *(3)*. This measures elastic constants by oscillating at frequencies of 10 to 6000 cycles per second. A modification allows measurements of shear which are recorded photographically.

We should decide which property we are most interested in measuring, shear or elastic modulus, or both, and select an instrument which is best suited for measuring this property. In measuring the quality of a fruit jelly we are perhaps most interested in shear. The instrument selected must not be unduly complicated, but rapidly adapted for routine use for control purpose. For research, especially with low-ester pectin gels, we certainly will need to use instruments measuring both shear and elastic modulus.

Since pectins lose grade on storage we should buy on the basis of grade for current deliveries but not be disappointed if we find some months later it does not conform to grade. We will also want to consider standards for color, clarity and solubility.

Because of the many diverse lines of specialized uses we are not ready for standardization of pectin for food use other than by preservers.

It is not within our province to consider standardization of pectin for pharmaceutical purposes but mention should be made that pharmaceutical pectin is a standardized product meeting specifications stated in the Eighth Edition of the National Formulary.

Low-ester pectins are new products and have not yet developed to the point where stabilization by standardization is considered desirable.

Grade Standards have been developed by the Agricultural Marketing Service for such ill-definable products as a number of fresh fruits and vegetables both for the fresh market and for processing. An exception is the cider apple—no one is willing to define it on paper. For the past two months the Food and Drug Administration has been conducting hearings with a view toward defining standards for certain frozen fruits. It is estimated that with the different varieties and various methods of packing over 300 products are under consideration. Surely we food technologists can determine and agree to standards for preserver's pectin.

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Notes and Letters

Survey of Food and Nutrition Research*

(Condensed)

In 1946, upon request of the Quartermaster Food and Container Institute for the Armed Forces, the National Research Council undertook to design and effectuate a nationwide survey of active research in the fields of foods and nutrition. The objectives of the survey were to be as follows:

(1) The determination of the major food research problems in the United States, particularly those of definite significance to the national welfare.

(2) A listing of facilities available for food research and development.

(3) An estimate of the extent of food research and a characterization of the subject matter covered.

(4) An evaluation of the adequacy of coverage in the various fields of food research.

(5) An evaluation of the adequacy of educational programs pointed to the training of food research personnel, both at undergraduate and graduate levels.

It was envisioned that the attainment of these objectives would not only serve the interests of national security but would also serve as a convenient basis for coordination of research and guidance for all concerned with foods and nutrition.

The task of implementing the survey was assigned to a committee of the Food and Nutrition Board which operates within the Division of Biology and Agriculture of the National Research Council. The committee was constituted of Dr. C. G. King, chairman; Dr. Floyd Daft, Dr. C. A. Elvehjem, Dr. H. E. Longenecker, Dr. L. A. Maynard, Dr. H. E. Robinson, and Dr. Louise Stanley. Mrs. Mildred Ragan was engaged as technical secretary for the committee.

As the first step, a listing of academic, industrial, and governmental laboratories engaged in food and nutrition research was compiled, and this was extended to include those who financed research outside their own organization. This resulted in a compilation of 522 universities or colleges, 1,005 industries, or trade associations, and 119 foundations, public trust funds, or research institutes concerned with food and nutrition research.

In August 1947 and in subsequent appeals the directors or heads of these laboratories were requested to furnish the following information:

(1) A list by subject of research projects pertaining to foods and nutrition currently active in or being supported by their organizations.

(2) A list of professional personnel responsible for conducting or directing the research listed.

Definition of the information desired was not explicit except to indicate that all phases of food and nutrition research were to be considered including chemical, physiological, technological, microbiological, and basic economic studies. Concepts as to the scope and nature of research vary widely. The committee felt it was better to allow those from whom the information was solicited to formulate the returns in accordance with their own understanding. In this way it was anticipated that limiting patterns and definitions would more or less establish themselves.

There were some misgivings, particularly among the larger industries, as to the intent and purpose of the survey. Also some difficulties were encountered in attempting to distinguish between so-called basic and developmental research. No clear-cut distinction can be made in this respect, and it was left to the judgment of those submitting the information as to what limitations should be applied. Likewise, some were reluctant to list their professional research personnel without a clearer indication of what kind of personnel list was to be drawn.

In spite of these and many other problems which came up while the information was filtering in, it is believed that a major portion of food and nutrition research active during 1947 has been recorded in this survey. In the 306-page compilation, published by the Food and Nutrition Board of the National Research Council, there are listed by subject over 4,000 projects in food and nutrition research. These were submitted from over 600 organizations. Of this number of organizations, 39 percent were classified as academic, 14 percent were classified as governmental, and 47 percent as industrial. The number of investigators listed approximates 5,000.

The initial report has been divided into four sections. The first section includes the list of numbered research projects classified by subject and referred to the appropriate conducting or supporting organization by number. The second section is an alphabetical list of organizations conducting or supporting food and nutrition research. Each organization is given a numerical code number to which the projects and personnel are referred. The third section is an alphabetical list of research personnel responsible for the conducting or direction of the research. The highest academic degree is included, and each investigator is referred to his organization by the assigned code number. The fourth section is a comprehensive index of the subject material referring to the projects by number.

In order to indicate the relative distribution of projects, the projects assigned to the field of physiology represented 40 percent of the total, projects involving primarily food chemistry made up 30 percent of the total, projects of food technology 15 percent, economics 9 percent, microbiology about 5 percent, and psychological studies numbered less than 1 percent of the total.

* Presented before the Eighth Annual Institute of Food Technologists Convention, Philadelphia, Pa., June 7, 1948.

This survey could probably be more properly designated as a comprehensive compilation or registry of food and nutrition research. It is hoped to keep the registry continuous and up to date. Appreciation is due those who have provided the information, and continuous cooperation is requested. There were unquestionably unfortunate omissions which it is hoped will be corrected. Copies of the published report and further information can be obtained from the Food and Nutrition Board, 2101 Constitution Avenue, Washington, D. C.

LeRoy Voris

Executive Secretary, Food and Nutrition Board, National Research Council, Washington, D. C.

Position of Food Technologists in National Emergency[a]

(Condensed)

That we were unable to strike back at the outset of World War II is a matter of record. Of particular concern to us was the relative unpreparedness of the food industries and of technologists, as a group, when the first blow was struck. What was wrong with the use we made of food technologists during the recent war? We have come a long way from the "Corn Willie and hard tack" diet of World War I. World War II moved us forward to such notable developments as rations with greatly increased variety of meat products, improved nutritive value, and greater stability in storage.

So much for the black side of the ledger; the red side is somewhat disturbing. Although industry, in peacetime, may have felt itself adequately supplied with food technologists, the special feeding requirements created by the national emergency staggered the industry with demands that could not be met adequately with the technologists available. These shortages of personnel were, of course, aggravated by the tremendous demands of selective service.

Another debit item, which in reality antedated the war, was the lack of a technological training program. Had systematic training programs been in operation, the nation could have adjusted itself to war much more rapidly.

But of all the items in the debit column, the most serious was the lack of proper utilization of our technologists by the Armed Forces. The report of the AD HOC Research and Development Advisory Committee of the Reserve Officers, dated March, 1948, presents some depressing facts. Out of a representative group of 100 technologists in uniform, the talents and skills of only 30 were fully utilized. The services and skills of 33 were utilized for only half of their period of military service. Out of the remaining 37, little or no use whatsoever was made of their technological education and experience. Such wastage is downright dangerous. Since the next war will be far more technological than the last, we must not let this happen again.

Guided missiles, armed supersonic planes, radioactive dust, or diabolical weapons could strike tonight.

Whenever they strike, however, tonight or in the future, we can anticipate two immediate problems—the serious disruption of normal food channels, and the total destruction of segments of our food manufacturing structure. As food technologists, we will be engaged in rehabilitating destroyed food facilities and in restoring normal food channels. We shall also have the *primary* responsibility of food research and development.

The problem of the food technologists will suddenly become two-fold:

(1) to insure adequate feeding of the domestic civilian population, and

(2) to provide rations for the armed services at home and overseas.

Let no one underestimate the complexities of the global feeding problem of the next emergency. The power of offense has been increased and warfare will be open and characterized by rapid movement. Airborne operations in a future war will probably be on a scope and magnitude not conceived of today. Many people, today, think war will be fought in all parts of the world with the likelihood that an important locus of operations will be the polar region. The implications are many. As for food, ration research and development must be pursued with renewed intensity, with particular emphasis on stability, acceptability, and nutrition. The rations must be compact, light in weight, adaptable to special environmental conditions, and easily prepared.

Added to these complexities is the need to adjust rations to the physiological and psychological requirements of soldiers under environmental and emotional stress. There will, of course, be many other problems.

After hostilities have once begun there will be little or no time for planning. The optimum moment for organizing our personnel and resources for total preparedness is now. We must think in terms of what will be expected of the food technologists in the next emergency. Further, there should be placed in the hands of the technologist a plan sufficiently detailed to indicate his individual responsibility.

The Department of the Army has drafted and approved an initial plan for the training and mobilization of scientific personnel who are Reserve Officers (per ltr OQMG 28 May 48) in a national emergency.

The objectives of the plan are:

a. To establish a program for Reserve Officers who are professionally engaged in research or development which will—

(1) Maintain the useful affiliation of this type of personnel with the Organized Reserve Corps.

(2) Provide peacetime assignment for these officers, enabling optimum utilization of their education, experience, and skills.

[a] Presented before the Eighth Annual Institute of Food Technologists Convention, Philadelphia, Pa., June 7, 1948.

(3) Furnish mobilization assignments which will fully utilize their talents.

(4) Adequately prepare these officers for mobilization.

b. To establish adequate means for the selection of Reserve Officers of this category to participate in this program.

The plan itself calls for an organization in each military district where there are a sufficient number of reserve officers who are professionally engaged in research and development. For example, the technologists of the Philadelphia area under this plan may be organized into a Reserve Research and Development Group, provided a minimum of 20 officers indicate their desire to be so organized. Where fewer than 20 reserve officers are available, sub-groups may be organized.

The final selection of qualified reserve officers for participation in this program will be made by the Department of the Army. In order to be eligible for selection, reserve officers must have the following qualifications:

(1) Possess at least a Bachelor's Degree, from a college or university accredited by one or more of the recognized national or regional educational accrediting associations.

(2) Be engaged in a professional capacity in research and/or development work in a field normally associated with one of the physical, engineering, medical, or biological sciences, including pertinent fields of psychology.

Professional employment in a field or activities more properly associated with routine or maintenance functions will not necessarily be considered as professionally qualifying an officer to participate in this program.

These requirements may be waived by the Department of the Army if the applicant's professional background and experience so warrant.

In peacetime, reserve officers will be given inactive duty assignments by the Department of the Army to Organized Reserve Research and Development Groups or sub-groups for training. Inactive duty training will have the following scope:

(1) Orientation on the organization of research and development activities of the Department of the Army, of the Army Field Forces, and of the various technical services, including those boards and panels whose functions fall within the purview of research and development.

(2) Pertinent parts of courses from established schools and colleges of the armed forces.

(3) Review and editing of technical manuals and publications.

(4) Work on certain research and development material and non-material problems as assigned by the head of the appropriate technical service in coordination with Research and Development Group, Logistics Division, General Staff, United States Army.

(5) Review or formulation of plans to assist research and development organizations, boards, or panels in accomplishment of their mission.

(6) Review or formulation of planning methods or the evaluation of proposed tactics, technics, and organization.

(7) Assistance to service schools in revising or broadening their training programs.

(8) In view of the technical qualifications of the Reserve Officers participating in this program, they will be encouraged to formulate their own training programs encompassing research and development problems or projects of interest to the Department of the Army.

Active duty training will be subject to the availability of funds. Reserve Officers assigned to Organized Reserve Research and Development Groups or Subgroups, may be assigned to research and development agencies, schools, and boards of the Department of the Army, the Army Field Forces, area commands, or the various technical services for active duty training upon request.

In time of war, assignments will be made to appropriate Research and Development installations or offices. These assignments will be made by the Department of the Army. Each assignment will be subject to review at any time to determine that the officer concerned is the best officer available.

That is the essence of the plan.

COL. C. S. LAWRENCE
President of the Quartermaster Board,
Camp Lee, Virginia

Neglected Aspects of Food Technology

All food technologists should aspire to be broad-gauged, fully-rounded members of the profession. It is imperative that all members of I. F. T., especially those of us who have left the institutions of learning and are now in industry, should endeavor to develop themselves to that end.

Nevertheless, one need only look at the programs of our meetings and the contents of our journal to observe the tendency of our interest to center around the preservation of fruits and vegetables. Which is not to object to this important interest but to point out that there is far more to food technology than the mere preservation of certain perishable foods by heat sterilization or freezing. We are decidedly lop-sided in our interests at present.

Those who have reached mature years in food technology are not looking for rapid transition from a narrow or highly specialized view of the profession, but they have a right to look for important nearby moves in the direction of broad fundamental training for those about to enter our ranks. It is our duty to encourage—or even insist—on adequate training for the coming generation of food technologists.

Is not the production of milled cereals within our field? Is not the production of bakery goods a part of food technology? How about dairy products of all sorts? Confectionery? Malt beverages? Carbonated beverages? Shortenings? Bread spreads?

Or, instead of considering the field, product by product, how do we stand on functional concepts? We are strong on science, especially chemistry, biochemistry, bacteriology and nutrition; we are strong on sanitation and certain aspects of heat application; we are fairly good on control of the oxidative deterioration of fats, also on quality control—to mention a few of our better points.

But we are weak on many other important matters. For example, we are weak on management, administration, plant layout, process engineering, packaging and materials handling. The latter pair are definitely within the province of food technology, although the matter of plant layout and process engineering is regarded by some of our best known food technologists as outside our scope. To the extent that industry does not need to turn to the food technologist for solution of such problems, the opposition view is undoubtedly right. There can, however, be no debate on the need to include packaging and materials handling in food technology. Too often the food technologist is lacking in a fundamental understanding of this inseparable pair of functions, yet he is under a definite obligation to include them in his professional equipment.

Perhaps there is an historic reason for the neglect of packaging and materials handling by the majority of those who now constitute our membership. Heat sterilization of foods requires the use of a rigid container—the tin can or the glass jar. Such containers happen to be the most durable consumer packages that exist, especially the tin can. Since food technology has achieved recognition more rapidly in the field that uses the tin can or glass jar than in any other, we are wont to accept the rigid container without realizing that it is a package. And certainly we accept it without realizing that among all types of packages these two types of rigid containers occupy a special—almost unique—position. They are very "long-lived" packages, and they provide more nearly complete protection than any other conventional package.

A further influence on our acceptance of rigid containers without conscious realization of their packaging function is the fact that the makers—not the users—of these containers always decide what gauge of steel, what weight of tin plate, what lacquer or enamel is appropriate for tin cans or for metal closures that a customer may need. The food technologist of the packer's organization does not specify these things very often. Usually he or his company receives them without knowing that the technical decisions have been made on his behalf by the container manufacturer.

What a contrast to the problem of the food technologist in dealing with flexible containers! Here he is largely on his own. He alone must decide what materials are needed or are satisfactory to package his product.

In all types of food the proper packaging is for a definite period of time, the length of which is determined by many factors such as: the characteristics of the food, the climate in which it is to be distributed and consumed, the sales and distribution policies of the food company, the sanitary environment to be withstood, the rapidity of turnover, and so on. The period may vary from a few days to a matter of a few years. It is a finite time. One does not package for eternity—it costs too much, even if it were technically possible.

Decisions on the preceding matters are usually executive decisions concerning which the younger food technologist may have no voice whatsoever. But the determination of the characteristics of a food that requires protection is definitely the responsibility of the food technologist. Corporation executives' views about packaging characteristics are frequently a trifle dilute.

Despite this responsibility for the preservation of food quality, the available tabular information of the needs for a food package, stated in terms of that food's packaging characteristics, is extremely limited. Doubtless this is due to the fact that the majority of present day food technologists have been concerned only with the rigid container (of long life) which has always been indispensable to heat sterilization. Thus the tin can and glass jar automatically obviate the need for study of many packaging characteristics. For we could not have had heat-preserved perishable foods without the rigid package.

As soon as we enter the field of foods suitable for being packaged in flexible containers we run smack into a host of problems of a much different nature, which today are solved almost entirely by trial and error. If food technologists—or shall we say food technology—were abreast of the needs of the day there would be systematic research on packaging characteristics, and a vast amount of data would be available to permit short cuts to packaging success rather than the time-honored and costly system of trial and error.

Materials handling cannot be logically separated from packaging, even though it is definitely within the field of mechanical engineering, for the reason that packages of food must be moved and handled after filling and closing. The selection of any package without due consideration of the correleated materials handling problems is often likely to be a more expensive choice than one based on an integrated knowledge of the two.

It is gratifying that a few of the institutions now teaching food technology are beginning to consider packaging in its proper place in food technology. All must ultimately do it. And those individuals who have already embarked in industrial careers must train themselves sufficiently so that their judgments will not overlook the final disposition of their products.

Admittedly, packaging and materials handling are only a part of the chain of indispensable attributes of a well-rounded food technologist, but they have not yet received the recognition or study that they should.

L. V. BURTON, *President I. F. T., 1941-42.*

Use of Ion Exchangers in By-Product Recovery from Pineapple Waste[a]

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Tests on using ion exchangers for recovering canning syrup and citric acid from pineapple cannery residue juice are described. Chemical data and an outline of the process are included.

Introduction

The introduction of stable high capacity ion exchange resins opened up new possibilities in the recovery of valuable by-products from cannery waste materials. The exchangers may be used to separate and concentrate constituents which are present in such low concentrations that they cannot be economically recovered by other methods. They may also be used to remove small quantities of impurities from the solution of a valuable non-electrolyte such as sugar. Both types of application are found in the operations on pineapple waste. In this instance the major component, sugar, is obtained in a relatively pure solution and is concentrated to a syrup which is used on the canned pineapple. In the same operation, citric acid is recovered from one of the regenerant solutions.

The pineapple waste consists primarily of the shell of the fruit and other inedible parts. Material which enters the by-product plant amounts to about 40% of the weight of the pineapple as received at the cannery. This waste is passed through screw presses which extract about 50 gallons of juice per ton of fruit processed. The pulp from the presses is dried and used for cattle feed. The by-product juice is screened to remove a part of the fibrous material which it contains. It is then heated to about 190° F. to precipitate heat coagulable material and after the addition of filter aide is filtered to yield a clear acid juice.

The by-product juice has a solids content of about 11%. These solids consist of about 75-85% sugars, 7-9% citric acid and smaller amounts of malic and hydrochloric acids, potassium, calcium, magnesium and proteinaceous compounds. This juice has a color which is slightly darker than that of beverage juice. The sugars are about half sucrose and half reducing sugars. This combination makes the recovery of any sugar by crystallization very difficult.

This acid juice has been used in the past as a medium for dissolving cane sugar to make up the cannery syrups. Since this use could provide an outlet for less than one-third of the supply, in some canneries the juice was decolorized with carbon and concentrated to yield higher density syrups that could be entirely used up on the pineapple pack. In other canneries the excess juice was either concentrated and sold for livestock feed or was discarded to the sewer. The carbon treatment fails to remove many of the impurities which are prejudicial to the production of a syrup of the highest quality. Some of these impurities are especially harmful in that they accelerate the development of off flavors during storage of the canned pineapple products.

The use of suitable ion exchange resins to remove the impurities from the by-product juice produces an excellent quality sugar solution which can be concentrated to a syrup that is far superior to those obtained by carbon treatment. One of the arguments which has been used by supporters of the carbon treatment in comparison with the ion exchange process is the fact that the carbon removes only a small fraction of the 15-25% of non-sugar solids in the juice. Since these solids are concentrated and added to the canned pineapple, they contribute to the density of the finished product. They therefore are utilized to replace an equal weight of sugar. The ion exchange treatment is more expensive than purification with carbon; however, the superior quality of the syrups obtained appears to be ample justification for the use of the process by the pineapple industry.

The early research work disclosed that certain problems are encountered in the use of ion exchange resins with by-product pineapple juice which had not previously been reported in treating other sugar-bearing solutions such as those of the beet and cane industries. The most striking of these problems was the fact that all of the cation exchangers tested suffered severe losses in capacity. These losses might amount to as much as 25% in the first cycle and would continue to build up during subsequent runs, although at a slower rate. This decrease in capacity was accompanied by a falling off in quality of the treated solutions. This difficulty was traced to the adsorption of compounds which were not removed by the usual acid regeneration. These compounds are dark colored and possess undesirable flavors. It was found that these troublesome impurities could be largely desorbed by treatment with alkaline solutions.

Another problem in developing the application of ion exchangers to pineapple waste was due to the losses in capacity shown by many of the anion exchangers. These losses were at a much slower rate than those described for the cation exchangers. However, in some cases they amounted to 25% in 100 cycles. This loss is probably partially due to the action of the reducing sugars present in the waste juice. The most stable anion exchanger found was one which had been developed to withstand the degradative action of reducing sugars. This exchanger is Duolite A-3 which is produced by the Chemical Process Company.

The ion exchange purification of filtered juice from pineapple waste can be carried out in two different ways. The acid juice may be treated directly and the citric acid recovered entirely from the anion regenerant solution or a part of the citric acid can be precipitated by lime before passing through the exchanger beds. The solubility of calcium citrate prevents the recovery of more than 60-70% of the citrate by lime precipitation.

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The lime which remains in solution after the precipitation and filtration increases the cation content by about 50%. This decrease in anion and increase in cation loads is advantageous from the standpoint of the initial plant investment since the cation exchangers are much cheaper than anion exchangers. It is also desirable from the standpoint of chemical requirements for the recovery of calcium citrate. The liberation of the citric acid from the exchangers requires about one equivalent of alkali and a calcium salt such as calcium chloride must be added to precipitate the citrate. The combined costs of alkali and calcium chloride are appreciably greater than the costs of lime for direct precipitation plus the additional sulfuric acid which must be used for the cation regeneration in operating on limed juice. Therefore, the process adopted included the lime precipitation of citrate before the ion exchange treatment.

Ion Exchange Process

The ion exchange process as carried out on the juice from pineapple waste consists of the following steps:

(1) Demineralization with two pairs of exchanger cells. This phase includes the sweetening on and sweetening off of the beds.

(2) Regeneration of the cation and anion beds in series with a sodium chloride solution from which calcium citrate is subsequently precipitated.

(3) Backwashing and draining of the resin beds.

(4) Alkaline rinse of the cation exchanger to remove adsorbed organic compounds.

(5) Sulfuric acid regeneration of the cation resin.

(6) Sodium hydroxide regeneration of the anion resin.

Since the calcium citrate precipitation and filtration are carried out hot due to the lower solubility of the citrate at high temperatures, the clear filtered juice is cooled in two spiral heat exchangers before entering the ion exchange cells. In the first heat exchanger it is cooled by the treated juice as it is being pumped to the evaporator supply tanks. The second heat exchanger is cooled by brackish water obtained from shallow wells.

The cooled juice is stored in a holding tank from which it is pumped through the exchanger cells. This tank receives not only all of the raw juice as it enters the plant but also the returns from all of the sample lines and the high Brix juice which is obtained on sweetening off an exhausted pair of cells.

The cooled juice is passed through two pairs of exchanger cells in the sequence, cation, anion, cation, and anion. The flow rate is varied depending upon the speed of the cannery operations. The flow rate is set on an automatic controller. The ion exchange cells are 10 feet in diameter and 10 feet high. They have a saran pipe under-drain system which is covered by slightly more than one foot of anthrafilt on which the exchanger beds rest.

The sweetening on operation is controlled by a density meter and the water from the cells passes into the sewer until its sugar concentration reaches 2° Brix. The purified solution above this concentration is diverted into the treated juice tank and is subsequently sent to the evaporator.

The first cation exchanger serves to remove the bulk of the inorganic bases. The effluent from this cell has a pH of about 2.0 at the start of a cycle. The potassium content of the juice is reduced by the first cation from over 1500 ppm. to less than 5 ppm. There is a slightly higher leakage of sodium. The break-through of the inorganic cations is quite sharp with sodium appearing in increasing quantities slightly before the potassium and calcium.

The removal of colored impurities by the first cation is much less complete than is the removal of inorganic bases. It is the removal of these compounds by the second cation exchanger that makes a double pass system desirable for the production of a good quality sugar syrup.

The first anion exchanger removes the liberated acids and also a considerable portion of the colored impurities. The anion exchangers remove a much larger portion of the colored compounds than the cation exchangers. However, if the cation exchangers have become fouled so that they are not efficiently removing color the anion beds cannot satisfactorily remove these impurities and a poor quality juice results. The pH of the anion effluent will vary from about 10 at the start of a run to about 5.0 at the end. This pH will vary with the amount of rinse water which has been used in the previous regeneration and is also affected by the type of organic acids which are present in the juice. With weakly held acids the pH may drop quite rapidly.

The second cation effluent has a pH between 3.0 and 3.5 and an acidity less than 0.01 N. The sodium and potassium are present only in traces.

The second anion effluent is almost free of colored impurities. Its low ash content is indicated by the specific resistance which is between 5,000 and 20,000 ohms. The pH of the second anion effluent is adjusted automatically by mixing in a small amount of second cation effluent. This step is taken in order to avoid heating an alkaline juice which would lead to a decomposition of some of the reducing sugars. The Brix of the treated juice averages about 8.0.

The juice cycle is controlled by the pH of the first cation effluent. The run is stopped when the pH rises to 3.4. The juice remaining on the pair is sweetened off into a sweet water tank from which it is slowly blended back into the raw juice tank. The sweetening off step is controlled by conductivity. This control is possible because the electrolyte content of the juice varies with the density. A conductivity control is not possible in the sweetening on operation since the electrolytes have been almost completely removed.

In the sweetening off all of the solutions below 3° Brix are discarded. However, the use of at least a part of these sweet waters is contemplated in the future. With the present operating conditions the losses in dilute waters approach 2% of the solids entering the plant. This loss is not entirely made up of sugar.

The treated juice is concentrated from 8° Brix to 25° to 50° Brix depending upon the cannery syrup requirements. These final syrups have a light yellow color. A quadruple effect tubular high speed evaporator